

PROGRAMMED SHRINKAGE DILATOMETER SOFTWARE

The PS dilatometer software operates the dilatometer under either standard temperature or programmed shrinkage (PS) control. Under PS, furnace power is adjusted based on specimen shrinkage. For sintering of powdered ceramics and metals, this has the advantage of generating temperature/time schedules which facilitate efficient densification. There are three consecutive windows associated with the software: the **Profile** window allows user specification of experimental parameters, the **Centering** window facilitates shifting the LVDT output, and the **PS Dilatometer** window displays the real-time progress of the experiment in both numerical and graphical forms.

Profile Window

Changes can be made to experimental parameters by clicking on the blue text associated with each item, typing in new values, and either clicking "OK" or pressing ENTER. The uppermost container holds general operation parameters. The data file must describe the full path, and should have the extension ".prn". Two data files are automatically saved after the experiment is complete. The "<datafile>.prn" file holds data in a format compatible with the Theta data-plotting routines and "<datafile>.prn" contains an ASCII-format file with the full data set in a different form. The *Parameter File* contains all of the parameters set on the last run. It is read in and used in the entries on the **Profile** window, so that only items which need to be changed have to be entered. The default (startup) parameter file is "c:\ThetaPS\Params1.txt", but the parameters from other parameter files can be called up immediately upon entering a new parameter filename.

The *Specimen Length* and *Reference Length* fields refer to the specimen which contacts the rod which in turn extends into the core of the LVDT, and the reference which contacts the rod which in turn extends into contact with the LVDT housing. The *Pt Thickness* field is used to subtract out the expansion effects of platinum foil used in contact with specimen ends and the pushrod or casing. If no foil is used, this must be set to zero. The *Expansion Max* and *Min* settings are used for display purposes to set the y-axis range during real-time plotting. The *Slump Protection* setting is intended for when the specimen slumps or melts. If the specimen contracts past this limit, the furnace is immediately shut off. If only standard temperature control is desired, the *PS Schedules* should be set to 0. This will cause the PS-related containers to be removed. In such cases, the cooling schedules do not apply, and both heating and/or cooling schedules should be programmed in the heating schedules category (see below). If PS is to be used, the *Heating Schedules* are the number of segments (*Heating Rate*, *End Temperature*, *Hold Time*) before PS is to begin. *PS Schedules* is the number of PS segments (*PS Rate*, *End Expansion*), and *Cooling Schedules* are the temperature controlled segments after PS is complete. If the numbers for these fields are increased, all of the schedule information corresponding to these segments must be entered into the appropriate container.

The *Reference* option buttons allow selection of single crystal sapphire (usually of a standard length, used for precise CTE measurements) and polycrystalline alumina. The latter reference may be preferable for PS since commercial alumina can be cut to the same size as the powder compact to be sintered. When the type B thermocouple type option is selected, a special routine is used to bring the furnace to 200°C before standard proportional-integral-derivative control takes over. Over a one a minute time interval, power is raised to a constant level specified in the *Startup Power* field. That power is maintained until furnace temperature exceeds 200°C. This is done since a type "B" thermocouple does not have an adequate voltage output below this temperature. The *Temperature Max* field should be set to protect the furnace from overheating. If this temperature is exceeded, the experiment will be halted.

When only temperature control is to be used during the experiment, only the fields in the *Heating Schedules* container need to be filled in. Otherwise, the *Heating Schedules* container is intended for the heating schedule required to heat the specimen into a temperature range where sintering will begin. Often, two segments are used for this purpose. The first segment may be rapid (e.g. 15°C/min) to raise

the temperature to ~100°C below which sintering begins. Then it may be desirable to have the second segment have a slower rate (e.g. 5°C/min), so that the transition to PS can be more controlled (see subsequent discussion). In the *Programmed Shrinkage Schedules* container, the *PS Rate #* and the *End Expansion #* fields will be automatically converted to a negative number regardless of the entry, since only a negative number would make sense for sintering. For multiple PS segments, the end expansion values must be cumulative, whereby the last *End Expansion* entry represents the total setpoint specimen shrinkage.

In the *PS Correction Parameters* container, if the *Zero Shrinkage Safety* option is selected, PS will be halted and the cooling schedules will begin if the specimen shows positive expansion during the PS period. This will serve as protection for samples which have sintered to full possible density prior to reaching the setpoint density. This function only becomes active after half of the total PS shrinkage has occurred, by which time control system fluctuations should have settled past the point where this safety might be triggered prematurely. If the *PS Termination* is set to *Setpoint*, PS will stop when the setpoint shrinkage reaches the specified final contraction (negative expansion). If the *PS Termination* is set to *Specimen*, and the specimen contracts faster than the setpoint, then cooling schedules will be initiated when the specimen has reached the specified final contraction. The *CTE field* accepts the best estimate as to the value of the coefficient of thermal expansion of the sintering specimen. This is used to correct the measured specimen contraction during PS for the contributions of thermal expansion via the temperature change during PS sintering. The *Max PS Temperature* field accepts a temperature entry which corresponds to the highest temperature which the furnace may reach under PS. If this temperature is exceeded, the system initiates the first cooling segment. The *Nosedive* field specifies the instantaneous temperature drop at the end of PS. This is useful when very specific densities are desired, where once the terminal PS density is reached, the specimen endures an interrupted quench to cool it quickly out of the range where continued sintering can occur.

If the *At End of Heating* option button for the *PS Start* field (in the *PS Correction Parameters* container) is selected, PS begins immediately at the end of the last part of the last segment of the heating schedules. If sintering is not occurring at this point, rapid furnace power (and consequently specimen temperature) adjustments will ensue until sintering at the specified contraction rate occurs. If the *Contraction Rate Match* option button is selected, then PS begins anywhere along the heating schedule when the specimen contracts at a rate equal to or greater than the setpoint rate of the first PS segment. This option often facilitates a smoother transition from temperature control to PS control. During initial heating, non-steady conditions may cause a falsely indicated specimen contraction which would activate PS too early. For that reason, the *Contraction Match Start* field allows a temperature entry, below which switchover to PS will not occur. This is often set ~100°C below the temperature in which sintering contraction is expected to initiate.

The *Cooling Schedules* container contains similar entries as those for the *Heating Schedules* container, and the different segments may be programmed for heating and/or cooling. *Cooling Schedules* is simply a designation for temperature-controlled segments after PS is complete. The temperature at the start of the first cooling schedule segment cannot be known ahead of time. Thus, the sign of *Heating Rate #1* will be changed as needed at the start of the segment to either cool to *End Temperature #1*, or heat to it, at the specified rate.

The fields within the *Control Parameters* correspond to proportional-integral-derivative control constants, separately set for temperature and PS control. Using temperature as example, proportional control adjusts furnace power proportional to the difference in temperatures between the sample and setpoint. Integral control adjusts furnace power based on the differences in setpoint and specimen temperatures integrated over time. Derivative control adjusts furnace power based on the differences in temperature (vs. time) slopes. These three control functions are added to establish the overall furnace power instruction.

The individual control contributions to the sum can be increased by: decreasing the constant (field value) associated with proportional, and increasing the constants associated with integral and derivative. The proportional constants are the "proportional bands", where if the sample temperature/expansion falls

outside of the proportional band, furnace power is set at its maximum extreme (either 0 or full power). The constants associated with each control parameter may need adjustment for widely varying schedules, but for similar schedules on different samples, they should not need to be changed. If the specimen temperature/expansion oscillates rapidly about the setpoint, the proportional constant is too small and should be increased. Conversely, if the control system adjusts furnace power only sluggishly to temperature/expansion behavior, the proportional constant should be decreased. If the temperature/expansion oscillates broadly about the setpoint, the derivative constant should be increased to force the temperature/expansion slope toward a constant value. If the control is highly jagged (furnace power jumps up and down) the derivative constant should be decreased. If the temperature/expansion behavior appears to show excess momentum (e.g. broad oscillations of increasing amplitude), the integral should be decreased. If the temperature/expansion behavior only slowly becomes coincident with the setpoint, the integral constant should be increased. The *DerPoints* field refers to how many of the most recently acquired points is the derivative to be calculated over. The more points used results in a more smoothly changing calculated derivative (slope), but one which is less representative of the most recent slope trends. Derivatives are determined based a the best-fit second order polynomial to the specified number of points, and then determining the slope of that function at a time corresponding to the most recently acquired point. The *PO* field corresponds to the startup power value. All furnace power adjustments from the control system are made relative to this seed value.

Clicking the start button will bring up the **Centering** window. If the data filename is already on the hard drive, a warning box will come up; if the same filename is then used, the previous one will be overwritten.

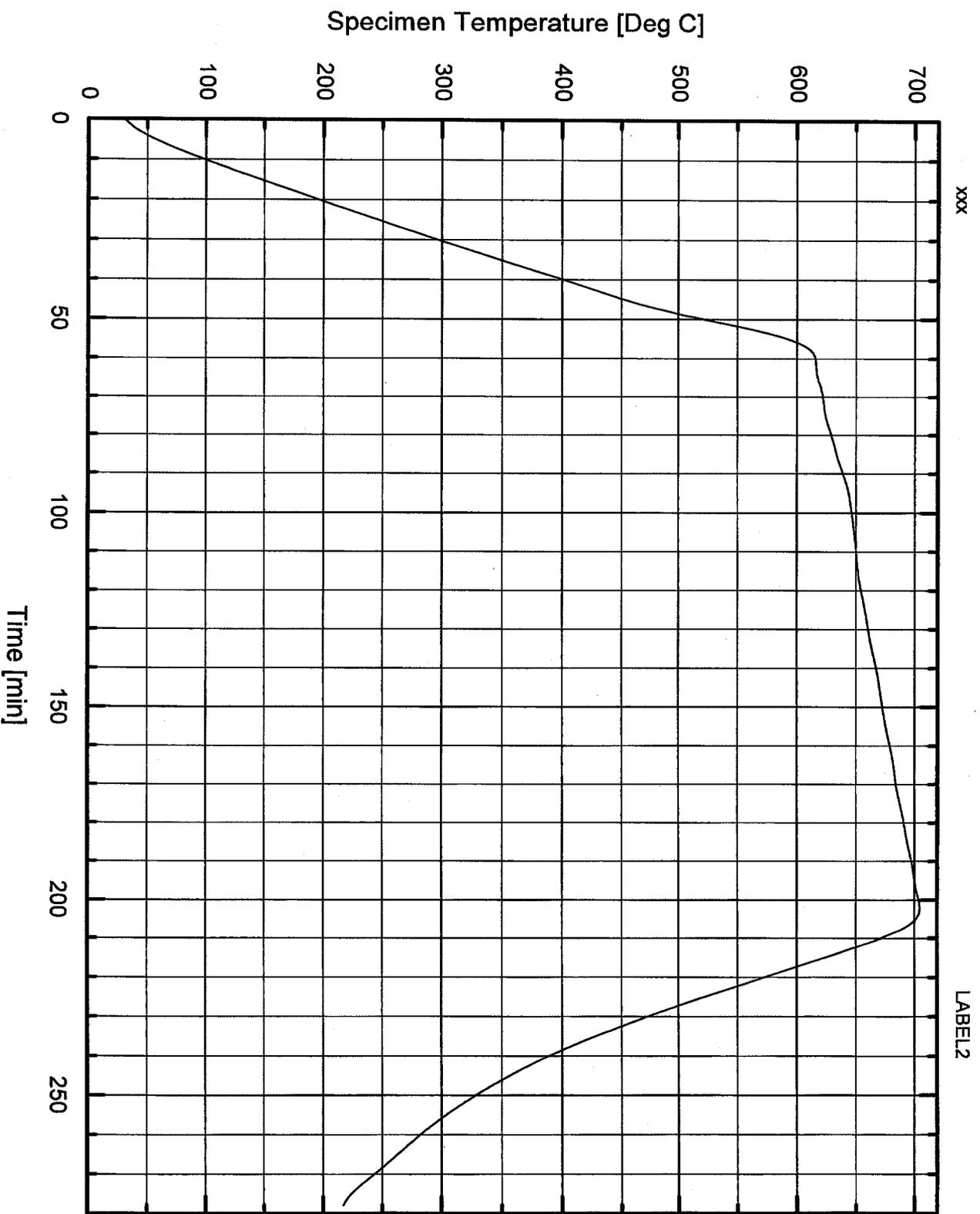
Centering Window

The centering window is an indicator of where the LVDT core is positioned relative to the LVDT housing. Specimen dimensions should be selected so that after contraction, the LVDT output is within its full scale range as well as the full scale range of the data acquisition board. The Theta LVDT stage has an electronic offset which should be exploited for this purpose by shifting the position of the marker so that there is plenty of room for contraction without exceeding the full scale limit.. The *Calibrate LVDT* button permits calibration of the LVDT output based on the micrometer on the LVDT stage. Instructions for use will come on screen after the button is clicked. This calibration is set by Theta and may need to be changed only rarely over time. Clicking the *Begin Experiment* button will display the real-time plotting screen and start the experiment.

PS Dilatometer Window

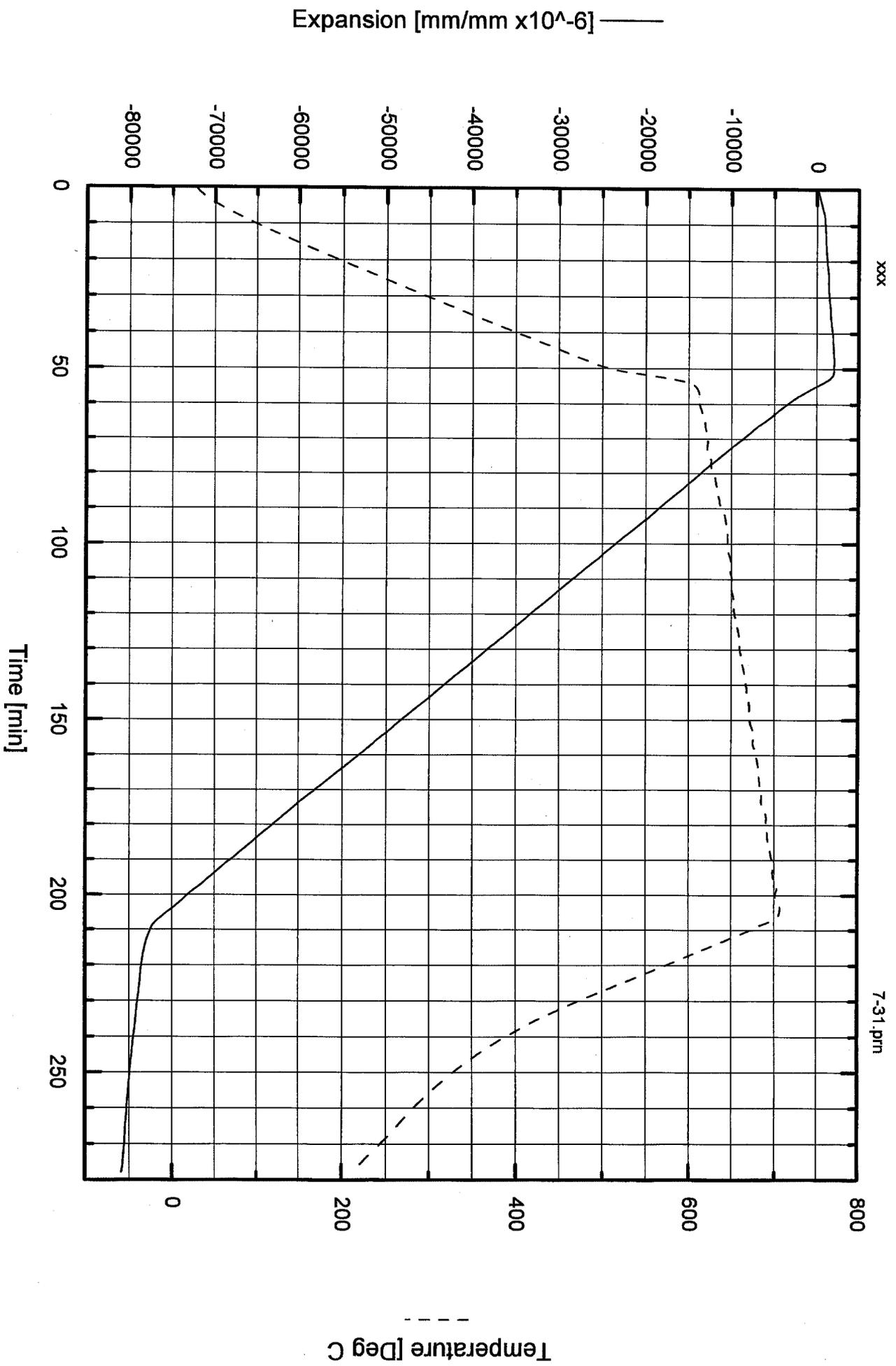
This window displays all pertinent information during the experiment in both numerical form near the base of the screen and in graphical form. The colored lines in the plot are as follows: specimen temperature--black, setpoint temperature--blue, specimen expansion--red, and CTE-corrected expansion during sintering--green. When PS segments begin, an additional text container appears with pertinent information. The run can be safely terminated at any time by clicking the stop button in the upper left hand corner, or by pressing ESC. Either will save the data collected to that point and shut the furnace off. An additional button labeled *Switch to Cooling* will appear during PS, which when clicked will initiate the first cooling segment.

Temperature vs. Time
ZnO Sample, 2.46 mm long, 7-31.prn, 7/1/00



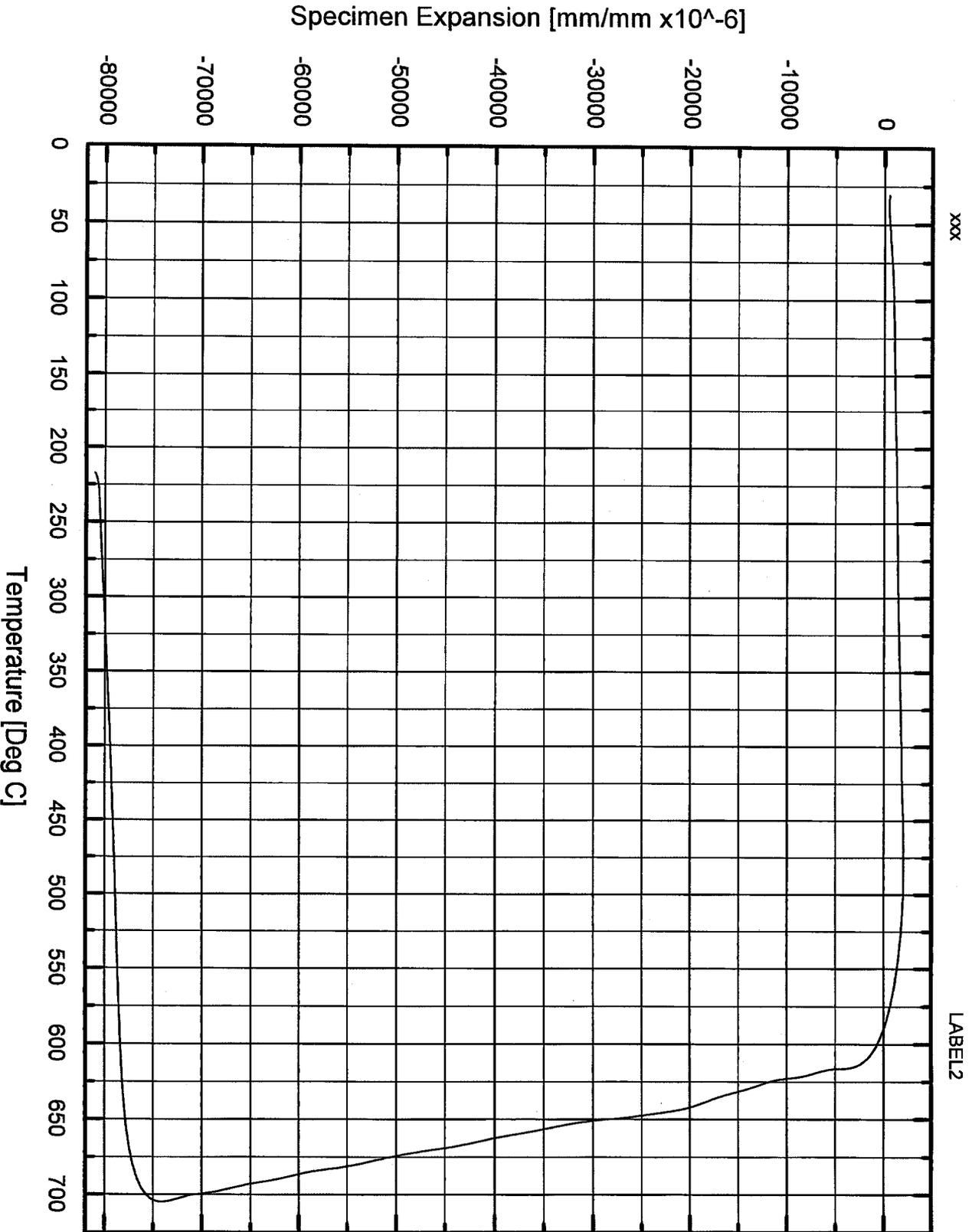
Heat 10 c/min, PID: 4-1.0-.2-5, PS. 6-2.0-200-20, smooth 3

Temperature & Expansion vs. Time
ZnO Sample, 2.46 mm, shrink 0.05%/min, 7/1/00



Heat 100C/min, PID 4-1-0-0.20-5-0, PS 6-2-0-200-20, Smooth 3

Specimen Expansion vs. Temperature
ZnO Sample, 2.46 mm long, 7-31.pm, 7/1/00



Heat 10 C/min, PID: 4-1.0-2-5, PS >6-2.0-200-20, smooth 3