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Dilatometry for Thermal Expansion Manuscript

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Overview

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Thermal expansion is extremely important when considering which materials will be used in systems that experience fluctuations in temperature. A high or low thermal expansion in a material may or may not be desirable, depending on the application. For instance, in a common liquid thermometer, a material with a high thermal expansion would be desirable due to its sensitivity to temperature changes. On the other hand, a component in a system that experiences high temperatures, such as a space shuttle re-entering the atmosphere, will need a material that will not expand and contract with large temperature fluctuations in order to prevent thermal stresses and fracture.

Dilatometry is a technique used to measure the dimensions of area, shape, length or volume changes of a material as a function of temperature. A principal use for a dilatometer is the calculation of thermal expansion of a substance. The dimensions of most materials increase when they are heated at a constant pressure. The thermal expansion is obtained by recording the contraction or expansion in response to changes in temperature.

Principles

Dilatometry is performed by first measuring the initial length of the sample by hand using calipers, and then measuring the length of the sample while it is subjected to specified temperatures for specified amounts of time, this measurement will be recorded by a sensitive gauge in the dilatometer. While the sample is being measured a purge gas will be flowing through the furnace; (argon, nitrogen, etc) this will provide consistent atmosphere conditions, as well to keep the sample from oxidizing with oxygen in the air. Next, the sample is heated to a specified temperature at a specified rate, and the changes in dimensions are recorded with a sensitive measuring gauge. The change in dimensions could be either expansion or contraction. Thermal expansion is then calculated by dividing the change in length (L) by the initial length of the sample (). This process yields the average linear thermal expansion of the material. Several measurements of each sample yields more accurate results.

Thermal expansion can be instantaneous (the slope of the length vs temperature) or average (net change in length over a temperature range). The value can either be linear if only length is measured or volumetric if the change in volume of the sample is assessed.

Dilatometry can be conducted through several methods. The Dilatometer in this experiment uses a vertical push-bar method. (Figure 1) The thermal expansion experienced by the sample is transferred to the displacement sensor by the connected rod. However, since the rod is also exposed to the high temperature in the furnace, it too experiences thermal expansion. Thus, the resulting measurement must be corrected.

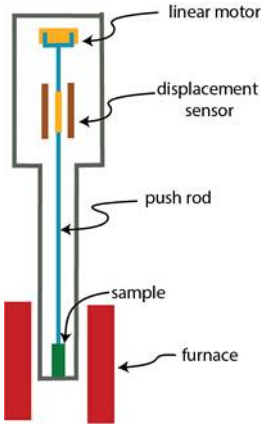

Figure 1: A schematic of a standard vertical push-rod dilatometer.

A comparable technology for measurement of thermal expansion is Michelson laser interferometry. The technique uses high precision lasers and mirrors to measure thermal expansion. Quality optics, photodetectors and interpolation techniques allow length resolution to about a nanometer. A unique feature of interferometry is the little restriction on size or shape of the sample. Another comparable technique is X-ray diffraction with the sample on a heated stage. Since X-ray diffraction can easily determine lattice parameter, it is possible to measure how the lattice parameter changes with temperature and extract a thermal expansion coefficient.

Procedure

1. Machine Start Up and Set Up. Begin with powering the computer, equilibrating the sample temperature ensuring it is at room temperature (about 20°C), and dilatometer on. Make sure cooling system is running and nitrogen gas is flowing along with all other necessary systems.

The nitrogen gas will need to be turned on between when the furnace is turned on and when the sample is inserted for testing. The pressure for the gas will be specific to the dilatometer, for ours it is 10 psi.

2. Determine which experiment will be conducted: calibration or expansion. For any set of expansion tests, a calibration test must be performed prior for reference. When doing an expansion test, select the most recent calibration that meets or exceeds your maximum temperature range and preferably is run at the same temperature ramp rate. When making a calibration for subsequent experiments, utilize a known standard. We will use a previously run calibration measurement of the known standard Crystallox. (Whether a calibration or expansion test is being run, the process for sample preparation, machine set-up, and establishment of parameters will be the same.)
3. Sample Preparation. For our experiment we will be testing a metal material. Accurately measure the length of the sample using high-quality calipers before inserting the sample into the furnace. Take several measurements along the length in order to establish measurement error. The sample should be long enough to allow the pushrod to exert some force on the top of the sample. If the sample is not tall enough, use spacers of a known material (measure the height of these so that the expansion can be subtracted from the results). The ends of the sample must be parallel within 1 degree.
4. Insert Sample. Clean the bottom surface of the furnace to ensure the sample has a flat place to stand. Lower the pushrod until it contacts the top of the sample. Lower the tube back into the furnace and ensure that the sample did not shift during lowering by checking the displacement gauge.
5. Establish Parameters. Follow ASTM E 228 standards for the material type. The important parameters include max temperature, heating ramp rate, dwell time, cooling ramp rate, number of repeats, and dwell time between repeats. Your parameters should match the calibration that you are using as closely as possible. Allow the temperature of the sample to reach equilibrium within the loading tube environment, at room temperature. The metal sample with be taken from a temperature of 20°C to 1000°C. Heat or cool at a constant rate equal to or less than 5°C/min. We will be not be doing any repeats in this test. The maximum furnace temperature for this device is 1200°C.
6. Verify Set Up. Before starting the test and walking away, double check that all systems are on and functioning, especially the furnace. Many dilatometers use a flow of nitrogen gas to keep the atmosphere of the test inert and constant, verify nitrogen purge gas is flowing.
7. Initiate Test. Start test and real-time data will be available for monitoring. If needed, the test can be canceled.
8. Save Data. Export and save data to the user's desired format, this will vary depending on how the data will be analyzed and presented. Typically, each sample should be run three times, with the first set of data discarded due to more significant expansion and contraction because of the thermal annealing of the sample.
9. Shut-Down. Verify all systems are powered down including the furnace, cooling system, and purge gas. Remove the samples from the furnace after ensuring that the furnace is cooled to near room temperature. Clean up workspace.
10. Analyze Data. Import data and create graphs and visuals to effectively represent your data.

Results

The results of dilatometers generally include data of temperatures, expansion lengths, and time. Different softwares used together with dilatometers can return results in different ways. Some softwares only return data points, while others have plotting functions and other analysis features. The software used in the procedure above used WorkHorseTM. This program returns data in a .txt file that can then be plotted using a software such as- Matlab, Qtgrace or Excel. **Figure 2** shows three different metals expanding and contracting as temperature is raised and lowered.

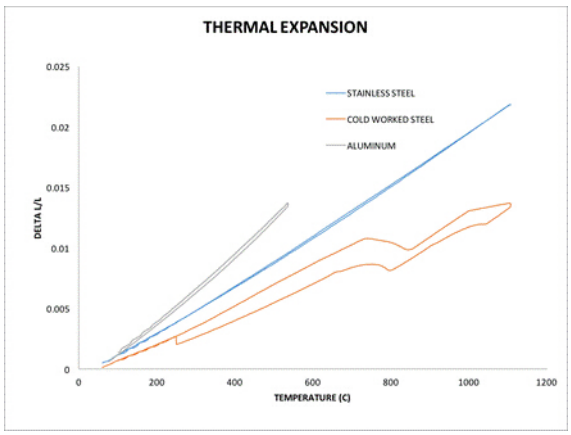

Figure 2: The change in length as function of temperature is plotted for stainless steel, cold worked steel, and aluminum. Samples are heated and then cooled with continuous length measurements to observe whether any hysteresis exists.

Thermal expansion is not always a linear function. This means that the coefficient of thermal expansion is not always constant. As seen in **Figure 2**, there is an unusual thermal expansion event that occurs in cold worked steel between 700°C and 900°C. In the case of stainless steel and aluminum the thermal expansion, as well as contraction, follow a linear slope. However, for the cold worked steel the expansion and contraction follows a non-linear change. This can be attributed to the dislocations in the cold worked steel. Locations where dislocation recovery occurs can experience different expansion/contraction as opposed to locations where normal expansion/contraction occur.

Applications and Summary

Dilatometry is a technique for measuring the dimensional thermal expansion of a material. Frequently this value is found by measuring the change in length as a material is heated and cooled. Thermal expansion is quantified by change in length divided by initial length. In addition

to thermal expansion, the technique offers insights into vacancy formation, phase changes, and dislocation evolution in response to heat treatments.

While determining thermal expansion of materials is a very popular use for dilatometers, there are other applications for them. For example, these instruments can also be used as a method to monitor phase changes in certain alloys. Determining dislocation densities is another application of dilatometry.

Monitoring Phase Changes: The application of dilatometry in phase change research is due to the change of the specific volume of a sample during a phase transformation. Lattice structure changes when a material undergoes a phase change. By recording the transformations taking place over a range of conditions, it is possible to present the results in a graphical form. This shows the formation temperatures of microstructural constituents that may be obtained for a given cooling or heating condition. This technique is widely used to study the transformation behavior of steels during continuous heating, cooling, and isothermal holding. This has immense value in metallurgical applications. It is important in engineering industries where steels are used for construction.

Dislocation Densities: Dislocations occupy a volume and therefore, as dislocation density changes in response to heat treatments, dilatometry can be used to observe and quantify dislocations. High resolution dilatometry has extended the technique to the study of texture changes and rearrangement and annihilation of dislocations related to the recovery and recrystallization process. High-resolution dilatometry, together with a model of isotropic dilatation and atomic volumes can be used estimate the dislocation density introduced in microstructures due to isothermal decomposition of austenite.