Simplest laboratory design of differential thermal analyzer

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Materials undergo structural and compositional changes due to phase transition, melting, crystallization, decomposition etc. These changes can be studied using DTA. It consists of measuring differential temperature ΔT (called signal) between sample and a standard material when both are heated or cooled at a constant rate. As the sample undergoes a phase transition it absorbs or emits heat and the sample becomes cooler or warmer than the standard material. It give rise to DTA peaks [1].

Figure 1: Thermocouple connected in differential mode and DTA pans drilled in steel block

Figure 1 shows the designing technique used to prepare the instrument. For placing test sample and standard material two cups were drilled in a same steel block to maintain uniform temperature throughout. It will minimize undesired signal drift. Two thermocouples connected in differential manner is the heart of the instrument. Here two Chromel-Alumel thermocouples are connected to produce differential temperature ($\Delta T = T_1 - T_2$) or DTA signal. These are fitted in a movable arm such that the tip of each of these TC's can move into two cups provided in the steel block (Figure 2).

Figure 2: Sample holder: Tips of the differential thermocouples are arranged to move into DTA pans through movable arm

A homemade Kanthal wound tubular furnace was used for heating purposes. DTA signal and temperature

sensor outputs were connected to temperature module of National Instruments data acquisition system, which provided a DTA plot directly on computer screen (Figure 3).

Figure 3: Test setup containing DTA and data acquisition system

Figure 4: Differential temperature (ΔT) versus temperature plot showing phase transition of AgI for heating and cooling cycle

Here we studied the β to α phase transition temperature of AgI and observed corresponding endothermic and exothermic peaks for heating and cooling cycle respectively. It was found very close to literature value (148°C) as shown in Figure 4.

The result was found quite correct. Associated energy can also be found by measuring the area under peak once calibrated with a calorimeter. Signal drift, as shown can be further minimized by using a precise furnace. Multimeter along with interfacing adapter can also be used instead of data acquisition system.

Reference

1. E.C. Subbarao, L.K. Singhal, D. Chakravorty and Masshal F. Merriam, Experiments in Materials Science.