Fabrication of low cost thermomechanical analyser (TMA)

S K Disawal*, U R Kapadi and D G Hundiwale*
School of Chemical Sciences, North Maharashtra University, P O Box 80, Jalgaon 425 001

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Fabrication of low cost thermomechanical analyser(TMA) which is one of the versatile instrument required for determination of glass transition temperature ($T_g$) of polymer is reported. The cost reduction during fabrication is so extensive that the entire cost of the instrument can be as low as Rs 6000/- which makes it in fact, affordable for any college.

**Keywords:** Thermal analysis, TMA, Glass transition temperature

**IPC Code:** Int. Cl.: G 01 N 3/62

**Introduction**

The work reported here pertains to fabrication of low cost instrument. Almost all of the analytical techniques that produce temperature dependent data may be classified as thermal techniques. Although it is believed that thermal analysis techniques include mainly, thermogravimetry and differential thermal analysis, there are several other thermal techniques that are extremely useful for studying certain types of chemical systems. Many of these techniques possess the potential for fairly wide use in the future. Some of these techniques are used to supplement or complement the TG or DTA data that are available, and hence yield another insight into the investigation under study.

One of the more conventional thermal methods available includes, Thermomechanical Analysis (TMA).

Thermomechanical analysis is a technique in which the deformation of a substance is measured under non-oscillatory load as a function of temperature, while being subjected to heating under a controlled temperature program. In this technique, mechanical properties like, expansion, softening, and tension of a specimen are measured as a function of temperature. The technique can provide useful information on the maximum working temperature of materials like, polymers and adhesives.

The cost of these instruments is very high. It must be mentioned here that virtually all syllabi of graduate/post-graduate courses in Physics, Chemistry, Material Science, etc., include thermal analysis as a major topic. Unfortunately the topic is only taught in the class without providing any demonstration to the students, because the cost of the equipment is beyond reach of the academic institutions. As a result, the students are deprived of the hands on experience and learning basic principles pertaining to it. Hence, it is thought that, if such instruments are fabricated at a cost affordable to colleges the students will be benefited at large.

**Instrumentation**

The conventional apparatus used for thermomechanical analysis of polymers consists of the following parts:

(i) Furnace (~ -150 to +200°C),
(ii) Displacement measuring device,
(iii) Temperature programmer,
(iv) Probe, and
(v) Recorder.

An attempt is made to replace the above said parts with most economical substitutes. The details of which are given below.

**1 Furnace**

A separate furnace was fabricated for this purpose. Since the working temperature range of the furnace was kept between –150 to +200°C the furnace was expected to have provision for pouring a coolant, i.e., liquid nitrogen (in the instant case), so as to lower the temperature of the furnace to –150°C. A small cylindrical block of aluminium with 50 mm diam and 75 mm height was used for fabrication of the furnace. A large hole (20 mm diam and 50 mm depth) was drilled at the center. A hollow cylindrical heater (2.5 mm thickness) was inserted in the hole. The hole was
big enough to accommodate sample holder, thermocouple, and probe assembly. The aluminium block was placed in a stainless steel cylindrical box with a clearance of 5 mm for pouring liquid nitrogen to bring down the temperature of the furnace to the desired level. For pouring liquid nitrogen, a funnel and copper tube were provided. Both were covered with non-conducting strip, was provided on the top of the furnace. The stainless steel box was insulated from outside using glass wool and non-conducting strips. This assembly was placed in another stainless steel box. The arrangement of the furnace is shown in Figure 1.

2 Displacement Measuring Device
In thermomechanical analysis the measurement of the extent of penetration by probe into the sample is important. The magnitude of penetration is often less than 1 mm and therefore a sensitive device is required to measure such a small change. A conventional device is Linear Variable Differential Transformer (LVDT), which converts mechanical displacement into an electrical output. However, LVDT is the most expensive part of TMA. In the present work, a low cost simple dial gauge thickness measurement device (Mitotoyo, Japan), was used for measuring the displacement. This substitution lowered the cost of the thermomechanical analysis, but not the sensitivity, as the least count of the dial gauge was 0.01 mm. The change was easily measurable and could be plotted vs the temperature of the sample. This resulted in saving to the tune of 95 per cent of the cost of LVDT. The temperature of the sample could be recorded at any instant from the display of the temperature programmer.

3 Temperature Programmer
The temperature programmer model BTC-9090 Fuzzy Logic Microprocessor based having autotuning facility was used. It was manufactured by Theta Controls Ltd, Pune. The feature of this temperature programmer is the facility of selecting rate of heating
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When a polymer sample with glass transition temperature ambient conditions was subjected to thermomechanical studies, there was no penetration of the probe into the sample. When heated at the glass transition temperature the sample became soft like rubber and allowed the probe to penetrate. Thus, when readings on dial gauge were noted below the glass transition temperature, while heating from time to time, no change was observed. On the other hand, when glass transition temperature was approached the dial gauge indicated the extent of penetration. When a plot of extent of penetration vs temperature was drawn, two distinct regions were observed, one below glass transition temperature and other one above glass transition temperature (Figure 2). Both the regions were approximately linear in nature and hence, to determine the glass transition temperature the intersection points of the tangents (drawn on the linear portion) was considered.

Results and Discussion

A thermomechanical analysis is extensively used for the determination of softening point (glass transition temperature) of amorphous polymers. For the purpose of calibration, four different commercial samples (PMMA, PS, PVC, PC) with known glass transition temperatures were selected. Each of the sample was in the sheet form. The rate of heating was at 2°C/min. The dial gauge readings were recorded as a function of temperature and were plotted. Tangents at appropriate position were drawn and the intersection point of the tangents were determined. The temperatures corresponding to the intersection points were noted and compared with the reported glass transition temperature. The comparison is given in the Table 1.

Thus the fabricated thermomechanical analyser provided results in conformity with reported glass transition temperature of polymers.

The earlier units of thermomechanical analysis consisted of the recorder as an essential part. In this work the recorder has been eliminated. It is important to note that, the entire assembly cost of thermomechanical analyser is less than Rs 6000/-, which makes it really affordable for any college.

Conclusions

The glass transition temperatures of polymers measured using fabricated assembly were in close

<table>
<thead>
<tr>
<th>Sample</th>
<th>Glass transition temperature °C</th>
<th>Reported</th>
<th>Observed</th>
<th>Percentage deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Poly methyl methacrylate (PMMA)</td>
<td>106</td>
<td>105</td>
<td>0.94</td>
<td></td>
</tr>
<tr>
<td>Polystyrene (PS)</td>
<td>101</td>
<td>100</td>
<td>1.00</td>
<td></td>
</tr>
<tr>
<td>Polyvinyl chloride (PVC)</td>
<td>82.5</td>
<td>81</td>
<td>1.81</td>
<td></td>
</tr>
<tr>
<td>Poly carbonate (PC)</td>
<td>146</td>
<td>145</td>
<td>0.69</td>
<td></td>
</tr>
</tbody>
</table>

from 1 to 200°C/min. The readability of temperature was 0.1°C.

4 Probe

A pointed stainless steel metal probe having 180 mm length and 5 mm in diam was used for penetration. A separate assembly, consisting of sample holder, probe, thermocouple, and dial gauge was designed in such a way that the whole system could be inserted easily into the cavity of the furnace. The arrangement was made to put weights on the probe. Initially the sample was placed and cooled to a desired temperature and then a probe with flat was rested on the sample. The other end of the probe was connected to the dial gauge.

Working of the Instruments

The furnace was heated at a uniform heating rate using the temperature programmer. At the glass transition temperature the sample softened and the probe underwent a penetration due to its own weight (and additional weight, if used), which was indicated by the dial gauge. The readings of penetration were noted from time to time. The temperature of the sample was measured by thermocouple.
vicinity with their reported magnitude. The entire fabrication cost is so moderate that any college can purchase or fabricate the instruments.

References
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*Author for correspondence*
Telephone: (91) 0257- 2258419
Fax: (91) 0257- 2258403
E-mail: dghundiwale@yahoo.com
*Dr A G D Bendale Mahila Mahavidyalaya, Jalgaon*