Investigating Relaxation and the Glass Transition with Sugar Glasses and Home-Built Apparatus

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Overview
Glass is one of the most common materials and its properties and processing are highly dependent on its viscoelastic melt behavior and subsequent relaxation. The purpose of this research is to develop and evaluate low-cost, experimental techniques for the quantitative investigation of glass relaxation and the associated glass transition behavior. Low temperature glasses made from sugars and artificial sweetener are examined. We compare results from our home-built Differential Thermal Analysis (DTA) apparatus with those taken with a commercial Differential Scanning Calorimeter (DSC). We also present visual model for the volume shrinkage during “test-tube annealing” both below and above the glass transitions.

Background: Glass forming materials have high viscosity in their liquid state and are slow to form crystals on cooling. When cooling from liquid these materials go through a transition range where the volume change slows down and becomes “frozen-in” when the rate of cooling exceeds the response time of the viscous liquid. Thus the final volume is cooling rate dependant. This can lead to internal stress in large parts which are not properly annealed or relaxed. The glass transition (T_g) reflects the temperature at which the glassy solid begins to exhibit the more liquid-like response of the supercooled liquid. The T_g is characterized by a step change in the slope of the Enthalpy (or Volume) vs temperature(T) curve. Both DSC and DTA measure the slope of Enthalpy vs T response and a typical curve is shown below. During aging the structure of glass can gradually relax to a denser, lower enthalpy state. A post T_g endothermic peak can be observed after the glass transition step of glasses which are aged (relaxed). The area of the endothermic peak is a measure of the relaxation.

Glass Samples: Sucrose & Corn Syrup glass (hard candy) and the artificial sweetener Isomalt (a sugar based alcohol), prepared in the kitchen laboratory at moderate temperatures (~155 and 170 °C, respectively) with glass transition temperatures ~ 30-40 °C.

Results

DSC T_g & Aging Dependence

DSC curves (above) show progressive development of post T_g endotherm with increasing aging time. Peak area and T_g are plotted vs aging time (below).

DTA T_g & Aging Results

Home-built DTA apparatus ($<100 in parts) shows similar T_g and aging effects as the commercial DSC.

Influence of Annealing Temperature on Uniformity of Volume Contraction

Samples were heated in test tubes to liquid (< 100 °) and quenched at various temperatures below and above T_g. Volume shrinkage at center, measured by meniscus(dimple) depth reaches a minimum at annealing (quench) temperatures slightly above the T_g.

Conclusions

- Isomalt and sucrose glasses exhibit considerable aging effects
- Demonstrated in both DSC and home-Built DTA
- Post-T_g endotherms increase logarithmically with increasing time
- T_g shows slight increase in T_g with annealing time in Isomalt
- T_g remains nearly unchanged in sugar glass
- Minimum volume change (Meniscus depth) occurs slightly above T_g in both glass systems.

Overall low-cost apparatus adequate to investigate relaxation and aging effects in our lower-temperature glass systems.

References

Sugar glass recipe from IMI website: http://www.lehigh.edu/imi

Acknowledgements

This work has been supported by IMI-NFG, Lehigh University through National Science Foundations (NSF) Grant : DMR-0844014. We also acknowledge useful discussions with Dr. Himanshu Jain, Dr. Raymond Pearson and Binay Patel, as well as the organizers of the David and Lorraine Freed Undergraduate Research Symposium, Lehigh University.