聚氯乙烯的热分析测试

TA measurements on polyvinyl chloride

样品 未增塑 PVC Trosiplast 3255 的注射成型板材

CI -CH₂-CH-

条件 测试仪器: DSC, TGA

坩埚: DSC: 40 μl 标准铝坩埚, 钻孔盖

TGA: 70山氧化铝坩埚, 无盖

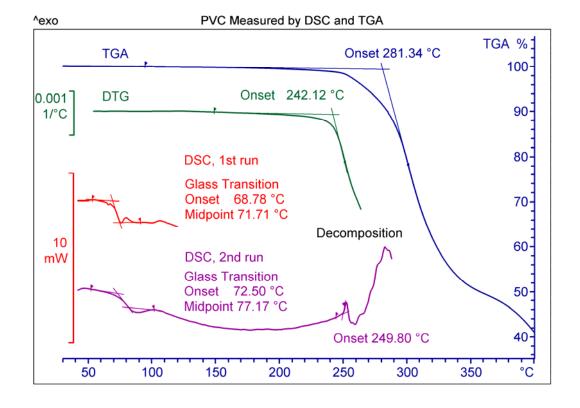
样品制备: 从板材上切下小片

DSC 测试: 以 10 K/min 从 30 加热至 120°C, 在自动进样器上骤冷

第二轮以 10 K/min 从 30°C 加热至 300°C

TGA 测试: 以 10 K/min 从 30°C 加热至 300°C

气氛: 氮气, 50 cm³/min



计算

玻璃化温度,起始点,第一轮,°C	68.8
玻璃化温度,起始点,第二轮,°C	72.5
DSC 分解起始点,°C	250.0
TGA 分解起始点,℃	281.0
DTG 分解起始点,°C	242.0

所有的起始点都基于拐切线。

解释 第一轮 DSC 曲线显示典型的不连续性。它们是由于应力释放和焓松弛造成的。 通常第二轮得到没有任何干扰的玻璃化转变。

在大约 200°C 以上,热降解通常开始,DSC 上为放热峰,TGA 测到的始终为由于 HCI 生成导致的失重。HCI 会侵蚀测试池。用氮气吹扫测试池是防止损坏的防范措施。如果只研究分解的开始,则可以中断温度程序,将仪器冷却下来。为了比较,第一个分解峰被计算作为起始点。

放热分解部分是由 HCI 与铝坩埚的化学反应造成的。

结论 PVC 是对热敏感的聚合物。它分解生成 HCI。

典型特征是玻璃化转变温度和降解开始温度(它是对热稳定性的量度)。

PVC measured by DSC and TGA

Sample Unplasticized PVC, Trosiplast 3255 injection-molded sheet material

Conditions Measuring cell: DSC,

TGA

Pan: DSC: Aluminum standard 40 μl, pierced lid

TGA: Alumina 70 µl without lid

Sample preparation: Disk cut off sheet

DSC measurement: Heating from 30 to 120 °C at 10 K/min, quench-cooled in the

sample changer

Second run from 30 to 300 °C at 10 K/min

TGA measurement: Heating from 30 to 300 °C at 10 K/min

Atmosphere: Nitrogen, 50 cm³/min

Evaluation

Glass temperature, onset, first run in °C	68.8
Glass temperature, onset, second run in °C	72.5

Decomposition onset DSC in °C	250.0
Decomposition onset TGA in °C	281.0
Decomposition onset DTG in °C	242.0

All onsets are based on the inflectional tangents.

Interpretation

The first DSC curve shows typical discontinuities. They are caused by stress re-lief and enthalpy relaxation. Usually the second run gives glass transitions free of any disturbances.

Above about 200 °C, thermal degradation often begins in the DSC with an exo-thermal peak and always with weight loss due to the formation of HCl, measured by TGA. The HCl could attack the measuring cell. Purging the cell with nitrogen is a countermeasure to prevent damage. If only the beginning of decomposition is to be studied, the temperature program can be interrupted and the cell cooled down. The first decomposition peak is evaluated as onset for comparison pur-poses.

Part of the exothermal decomposition is caused by the chemical reaction of the HCl with the aluminum pan.

Conclusion

PVC is a polymer that is sensitive to heat. It decomposes with the formation of HCl.

The characteristic features are the glass transition temperature and the temperature at which degradation begins (which is a measure of its stability toward heat).