

Thermal Analysis

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Analysis of Propellants by HyperDSC and TGA



DSC 8500

Introduction

Explosives, propellants and gun powders are often classed as highly energetic materials¹ and are a special subset of thermal analysis. Differential Scanning Calorimetry (DSC) is normally used to study these materials.² In this note, we investigate the application of HyperDSC™ and Thermogravimetric Analysis (TGA) techniques to slow-burn and fastburn gun powders. The instruments used are the Diamond DSC in its HyperDSC mode and Pyris™ 1 TGA, shown in Figure 1.

HyperDSC is a technique that involves heating and cooling samples at rates from 150 to 500 °C/min. Several papers have already addressed its applications.^{3,4} This work looks at its application to propellants as well as more traditional techniques.

Standard DSC

When gun powders or other energetic materials are run in the DSC, small sample weights (1-2 milligrams) are normally run at 10-20 °C/min. Figure 2 shows the results of such a run. It is important to keep sample size small and consistent in order to get reproducible results. On the below, for 5 runs, the temperature range was ± 4 °C. Enthalpy varied about 5% on repeated runs.



Figure 1. The DSC (left) and Pyris 1 TGA (right) represent the state of the art in modern thermal analysis.

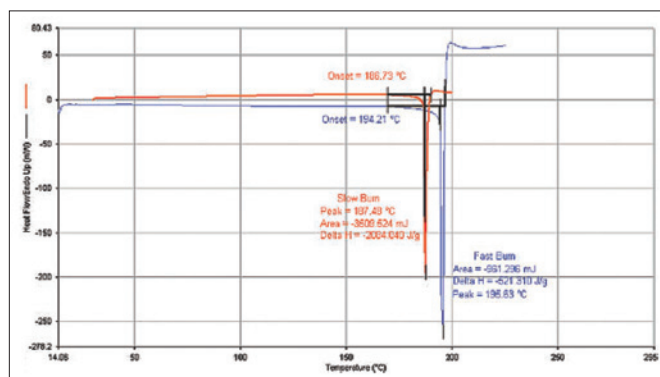


Figure 2. Slow-burn (red) and fast-burn (blue) gun powders run at 20 °C/min, N₂ purge and LN₂ cooling analyzed using a conventional DSC method.

HyperDSC

Using the HyperDSC approach gave similar results but at much faster turnaround times. Figure 3 shows the results for the analysis of the same materials using a HyperDSC method and scanning rate of 200 °C/minute. The instrument gave values for indium on recalibration at that rate which were within 1% of those seen at 20 °C/minute. Sample sizes were reduced to approximately a half a milligram and nitrogen was used as the purge gas. Samples showed similar behavior with variation for three samples being about ± 3.5 degrees and peak temperature was slightly shifted to higher temperatures. Variation in the peak temperature was similar to that seen in standard DSC while enthalpy showed about 8% variation. HyperDSC appears a viable option to increase the turnaround time for analysis of propellants with little or no loss in data quality.

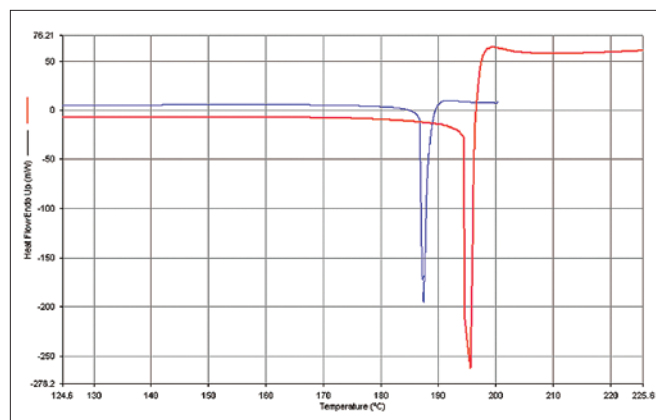


Figure 3. HyperDSC, run at 200 °C/min, on slow-burn (red) and fast-burn (blue) powders.

TGA

The high sensitivity of the Pyris 1 TGA and its ability to also heat quickly gave interesting results when running the two powders. Heating at 50 °C/minute gave the curves shown in Figure 4. The slow-burn powder shows more weight loss below 200 °C and both materials exhibit an abrupt weight loss at 209 °C. Both materials left a residue in the pans that did not burn off at elevated temperatures (1000 °C).

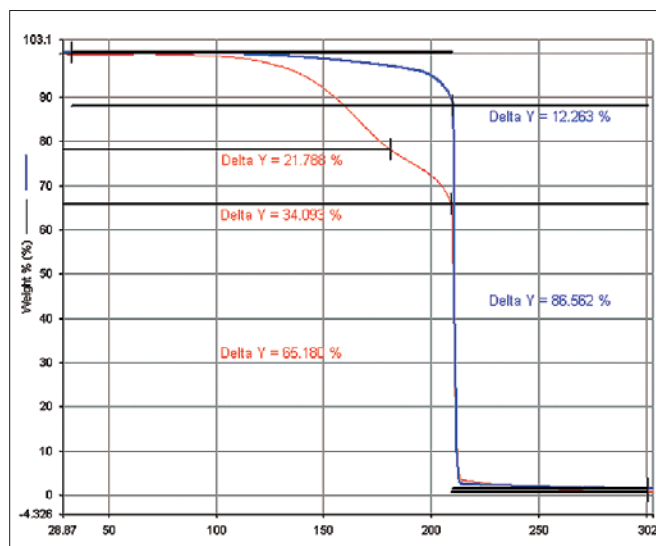


Figure 4. TGA on slow-burn (blue) and fast-burn (red) powders.

Conclusion

For quicker and improved analysis of propellants and other materials, HyperDSC allows dramatic improvement in turnaround time and the Pyris 1 TGA provides a more complete characterization.

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References

1. R. Rogers and J. Rogers, Explosives Science, Los Alamos National Laboratories, Los Alamos, NM, 2000.
2. J. L. C. Van Geel, Ind. And Eng. Chem. 58, 24 (1966). R. N. Rogers and L. C. Smith, Thermochemica Acta 1, 1 (1970). This shows some of the problems with programmed temperature kinetics methods. R. N. Rogers, Analytical Chemistry 44, 1336 (1972). Discusses isothermal rate constants. R. N. Rogers, "Differential Scanning Calorimetric Determination of Kinetics Constants of Systems that Melt with Decomposition," Thermochemica Acta 3, 437 (1972). R. N. Rogers and G. W. Daub, "Scanning Calorimetric Determination of Vapor-Phase Kinetics Data," Analytical Chemistry 45, 596 (1973). R. N. Rogers and G. W. Daub, "Determination of Condensed-Phase Kinetics Constants," Thermochemica Acta 9, 855 (1974). A. A. Duswalt, "The Practice of Obtaining Kinetic Data by Differential Scanning Calorimetry," Thermochemica Acta 8, 57 (1974). R. Bruce Cassel, "ASTM® Method of Testing for Determining the Arrhenius Kinetic Constants for the Screening of Potentially Hazardous Materials," PerkinElmer Thermal Analysis Application Study 28 [Pittsburgh Conference Paper No. 688, 1979].
3. T. Pijprier, V. Mathot, B. Goderis, and E. van der Vegte, NATAS Proceedings, 28, 32-37, 2000. T. Pijprier, V. Mathot, and B. Cassel, NATAS Proceedings, 28, 860, 2000. I. Platthaus, G.J. T. Laboratory, March, 2, 2002. B. Bilyeu, W. Brostow, M. Kesselman, and K. Menard, ANTEC Proceedings, 2003, 1878, 2003.
4. P. Gabbott, P. Clarke, T. Mann, P. Royall, and S. Shergill, A High-Sensitivity, High-Speed DSC Technique: Measurement of Amorphous Lactose, American Laboratory, August 2003.

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