STUDY OF PENTAERYTHRITOL TETRANITRATE SUBLIMATION BY ATOMIC FORCE MICROSCOPY

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Abstract: Sublimation of energetic materials is a critical process for their application and detection. Studies of sublimation using atomic force microscopy (AFM) allow one to monitor changes in the volume of individual microparticles and thus analyze compounds which are not suitable for classical research being too dangerous in handling or decomposing before sublimation under ambient conditions. However, both sample morphology and experimental conditions could significantly alter the results of the AFM study. The authors have studied in detail the effect of these factors alone with the substrate material influence on the correct assessment of the enthalpy of sublimation using AFM. The authors have observed the mechanical influence of the AFM probing process itself and the thermally-induced morphological changes of pentaerythritol tetranitrate (PETN) particles being heated at probing. Simultaneously with the PETN particles sublimation, at 45–60 °C, the increase in the particles volume was found indicating the recrystallization process which distorts the measurement results. However, on the mica substrate, the recrystallization process was not observed and the enthalpy of PETN sublimation could be correctly estimated from AFM. Additionally, it was found that for the correct measurement of the sublimation enthalpy of PETN, the particle height should exceed 280 ± 30 nm. In general, very small sample masses (< 10 μg) and relatively low temperatures used in AFM analysis allow safely obtain the correct sublimation enthalpy data for highly sensitive or low-volatile thermally stable materials and for the newly synthesized in small quantities energetic compounds with unknown properties.

Keywords: atomic-force microscopy; scanning probe microscopy; sublimation; PETN; energetic materials

DOI: 10.30826/CE23160411 **EDN:** QTTXML

Figure Captions

Figure 1 Thermally-induced morphological changes of PETN on Al_2O_3 substrate

Figure 2 Changes of volume (*a*) and volume change rate constant k (*b*) with temperature for PENT on mica (filled signs) and gold substrates (empty signs)

Figure 3 Mechanically-induced morphological changes of PETN on Si substrate: (*a*) crystallization of amorphous PETN in the scan area; and (*b*) destruction of the PETN particles and displacement of the fragments to the edges of the scan area

Figure 4 Changes of the PETN sublimation enthalpy estimated by AFM with material of the substrate (*a*) and particle height (*b*)

Table Captions

Table 1 Morphology and average size of PETN particles d_m on different substrates

Table 2 Experimentally measured average roughness R_a and contact angle θ and literature data on heat capacity C_p at 298,15 K and thermal conductivity λ of the substrates

Table 3 Literature data on PETN sublimation enthalpy

Acknowledgments

Ministry of Science and Higher Education of the Russian Federation is acknowledged for the financial support of the work (project No. 075-15-2020-803-P to Zelinsky Institute of Organic Chemistry).

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Received November 20, 2023

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