

Preparation and effects on stability and electrostatic sensitivity of α -AlH₃ by coated with stearic acid

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Abstract

The α -AlH₃ was coated with stearic acid (SA) by the method of solvent-nonsolvent. The crystal structure and the morphology of the prepared samples before and after coating were characterized by Fourier transform infrared spectroscopy (FTIR), elemental analysis (EA), scanning electron microscopy (SEM), X-ray diffraction (XRD) and X-ray photoelectron spectroscopy (XPS). The electrostatic sensitivity of α -AlH₃ samples before and after coating was measured by JGY-50 III. The results show that coating agent is firmly coated on the surface of α -AlH₃. The testing results show α -AlH₃ coated with SA can decrease the electrostatic sensitivity. The spark energy required for 50 % initiation probability (E_{50}) increased from 367 mJ to out of range.

Keywords: α -AlH₃, solvent-nonsolvent, stearic acid (SA), electrostatic sensitivity

1. Introduction

Aluminum hydride (AlH₃) is a promising hydrogen storage material due to its high theoretical hydrogen storage capacity (10.08 wt %), high volumetric densities (1.49 Kg H₂ m⁻²) and low decomposition temperature (100~200 °C)¹⁻³. Aluminum hydride (Alane) is an interesting material for a wide variety of application^{4,5}. It has been used as solid-state hydride in the solid rocket propellant by aerospace industry. Compared with Al, the specific impulse value of propellant with AlH₃ is higher in solid, liquid and solid-liquid propellants⁶⁻¹⁰. However, AlH₃ is composed of two atoms with strong reduction¹¹⁻²⁶, hence the problem of higher electric spark sensitivity remain unresolved, which poses great hazards for handling, transportation and utilization of this material^{27, 28}.

The three ways are surface passivation, surface coating and doping other substances. Surface coating has been the main method used for reducing the ESD sensitivity of an energetic materials. The appropriate coating materials and the coating processes could be selected according to different needs²⁹⁻³².

2. Experimental

2.1 Materials

Alane crystal used in this study is the α -AlH₃ and hexagonal crystals, which contain 9.80 wt. % hydrogen density. the α -AlH₃ is produced by Xi'an Modern Chemistry Research Institute. The coating material used is stearic acid (SA) from China Xi'an chemical group. Absolute ethanol from China Xi'an chemical group was used as solvent.

2.2 Coating process

The SA coating on α -AlH₃ involves several steps. First, the ethanol solution of SA was prepared with 0.03 g SA dissolved in 50 mL absolute ethanol. The content of SA added is 1 % m m⁻¹ (mass/mass percentages). Second, the α -AlH₃ (3 g) was dispersed in deionized water (50 mL) under ultrasonic irradiation. And then, the ethanol of SA was added into the above mixed liquor (α -AlH₃ with water). Lastly, the solution was filtered and the sample was dried at 40 °C for 2 h.

2.3 Characterization

The structure characterization of the α -AlH₃ and SA/ α -AlH₃ samples were performed by powder X-ray diffraction (XRD, DMAX2400 with Cu-K α radiation at λ =1.5418 Å). The morphologies of samples were examined by field emission scanning electron microscopy (SEM, Quanta 600FEG). FTIR spectra were measured with a Bruker Tensor 27 instrument (KBr pellets). Elemental analysis (EA) were obtained on PE-2400 equipment. The surface composition and chemical binding energies were analyzed by X-ray photoelectron spectrometer (XPS) with monochromatized (1486.6 eV)

2.4 Electric spark sensitivity test

According to GJB5891.27-2006 a standard test used to determine firing energy required to achieve 50 % probability of producing electric spark, the electric spark sensitivity test was evaluated by using Electric Spark Sensitivity Test Chamber (Model JGY-50III)³³.

3. Results and discussion

3.1 X-ray diffraction

The X-ray diffraction (XRD) analysis images of the α -AlH₃ (a) and SA/ α -AlH₃ (b) are shown in Figure 1. The XRD pattern of α -AlH₃ (a) in Figure 1 (2θ =27.84°, 38.58°, 40.72°, 46.1°, 49.96°, 57.26°, 63.26°, 66.26°, 68.14°) is compared with Ref. Code 00-023-0761(α -AlH₃)²²,³³, which corresponds to the crystal facet parameter of (012), (104), (110), (202), (024), (116), (112), (214). All the diffraction peaks were indexed to alpha crystalline and SA was amorphous. Because the SA layer is thin and the SA is non-crystal, there is no diffraction of SA in Figure 1 a.

3.2 SEM analysis

The microstructure of the samples investigated by SEM image of α -AlH₃ is shown in Figure 2a. SEM image of SA/ α -AlH₃ is shown in Figure 2b. The morphology before and after α -AlH₃ coating showed a typical crystalline form. Figure 2b shows that there is a thin film covering on the α -AlH₃ particles. The coating of SA is distributed uniformly in the picture that is magnified 2000 times.

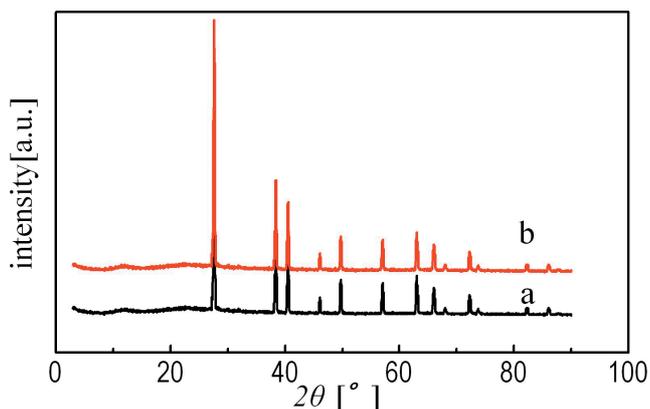


Figure 1 XRD patterns of α -AlH₃ (a) and α -AlH₃ coated by SA (b).

3.3 FT-IR Spectroscopy

Figure 3 shows the FTIR Spectra of α -AlH₃ (a) and SA/ α -AlH₃ (b). As shown in Figure 4, the characteristic vibration observed in spectrum (a) at 675, 866, and 1719 cm⁻¹, which correlate with the experimental data previously reported by Xing Wang Cai³³, are attribute to the α -AlH₃. In Figure 4, an additional band found at 675 cm⁻¹ can be attributed to the H-Al-H deformation mode in spectrum (a).

Additional bands corresponding to the α -AlH₃ spectrum were observed for SA/ α -AlH₃. There are two stretching vibration absorption peaks of C-H at 2918 cm⁻¹ and 2849 cm⁻¹. The position of the C-H band in the SA/ α -AlH₃ sample are close to the absorption for SA. These observations indicate the presence of SA.

New bands are not produced between the α -AlH₃ and SA which leads to the conclusion that it is a physical coating between the α -AlH₃ and SA.

3.4 XPS analysis

In order to determine the elements on the surface of SA/ α -AlH₃, XPS measurements were performed. The survey spectra are presented in Figure 4. In Figure 4 the main

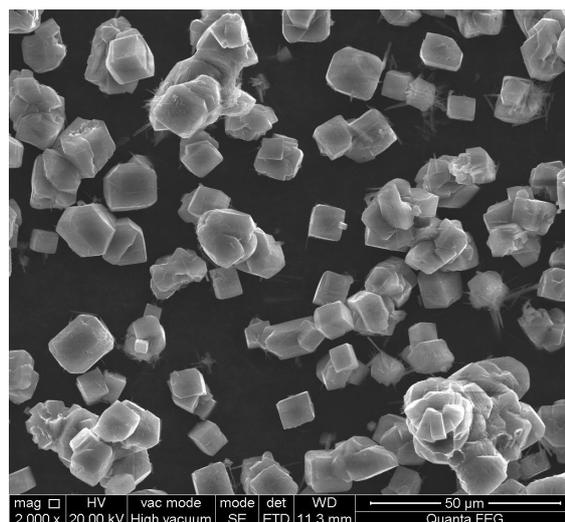


Figure 2a SEM image of α -AlH₃.

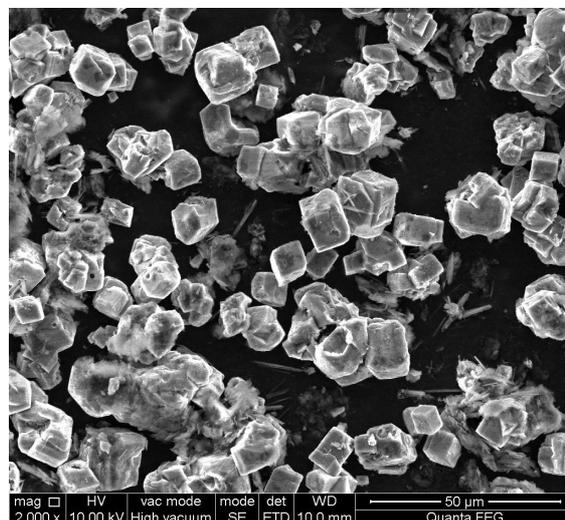


Figure 2b SEM image of SA/ α -AlH₃.

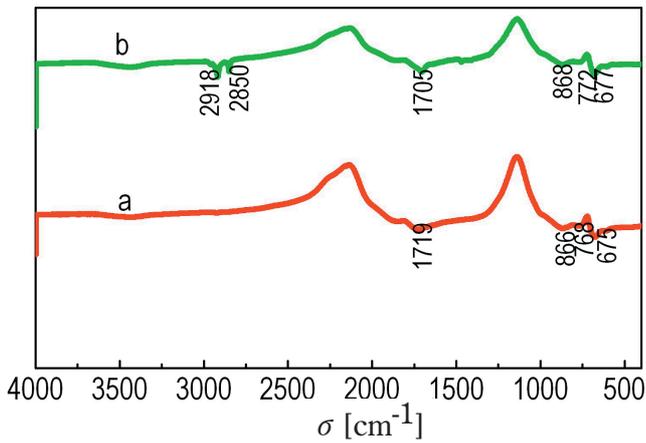


Figure 3 FTIR spectrum of the α -AlH₃ sample (a) and FT-IR spectrum of prepared sample SA/ α -AlH₃ (b).

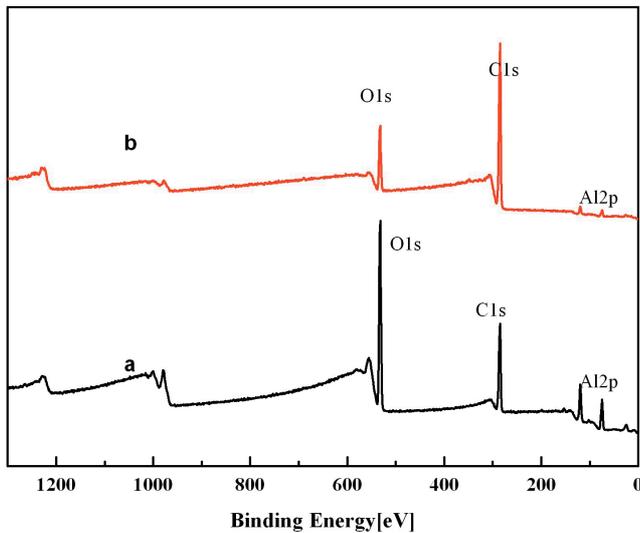


Figure 4 XPS patterns of α -AlH₃ (a) and SA/ α -AlH₃ (b).

peak centered at 284.6 eV corresponding to the standard binding energy arising from adventitious carbon has been used for the calibration of the XPS spectra. Apart from small contaminations by carbon, contributions, from only O and Al are observed. The XPS survey spectra of SA/ α -AlH₃ represent mainly C, O and Al contributions but also C and O contaminations. These observations are indicative of the presence of SA on the surface of α -AlH₃. However, several differences were found among the XPS survey spectra of SA/ α -AlH₃ and α -AlH₃. It should be noted that the peak intensity of Al_{2p} and O_{1s} decreased with the peak intensity the C_{1s} increasing. The phenomenon implies that the surface of α -AlH₃ is coated with SA successfully.

3.5 Stability analysis

The thermal stability analysis of α -AlH₃ before and after being coated by SA were studied by testing changes in hydrogen contents. The tests results are shown in Table 1.

From the above Table 1 we found that at room stored 365 days, the decomposition rate of α -AlH₃ is 1.1 %, but the decomposition rate of α -AlH₃/SA is 0.7 %. These results demonstrate that the coated α -AlH₃ decomposed into Al is more difficult than α -AlH₃ before coating. That is, the stability of coated α -AlH₃ increased.

Table 1 Changes in hydrogen contents of α -AlH₃ after long-term storage at room temperature.

No	Sample	Storage time [d]	Percent hydrogen [%]		Decomposition rate [%]
			before	after	
1#	α -AlH ₃	365	9.747	9.632	1.1
2#	SA/ α -AlH ₃	365	9.436	9.368	0.7

Table 2 Testing results of electrostatic sensitivity of α -AlH₃ before and after being coated by SA.

samples	Capacity [μ F]	Positive		Negative	
		V_{50} [kV]	E_{50} [m J]	V_{50} [kV]	E_{50} [m J]
AlH ₃	0.1	2.71	367	2.66	354
SA/ α -AlH ₃	0.22	–	–	–	–

“–”out of range

3.6 Electric spark sensitivity analysis

The testing results of electrostatic sensitivity of α -AlH₃ before and after being coated by SA are shown in Table 2.

From the above Table 2 we found that after 1 % SA is coated on α -AlH₃, the E_{50} value increases to out of range. This value is much higher than the maximum energy. The maximum energy is the power detection limit of the apparatus. The results indicate that the SA/ α -AlH₃ electric spark sensitivity decreases compared with that of α -AlH₃, and these results could be attributed to coating of the SA, indicating that SA/ α -AlH₃ is safer for electrostatic discharge.

4. Conclusions

The α -AlH₃ is successfully coated by via solvent-nonsolvent method. The characterization of the composite sample indicates that SA coated on the surface of α -AlH₃ uniformly. The novel SA/ α -AlH₃ composite exhibited considerably low decomposition rate and low static spark sensitivity. Thus, the novel materials used in this work might be suitable for the coating of a wide range of hydrogen and energetic materials, helping build the gap between low sensitivity and high performance. Future work will be directed towards choosing suitable coating methods and optimization of process parameters.

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