Preparation and Characterization of Aluminum Hydride

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Abstract: α-AlH_3 was prepared by organometallic synthesis route. The characterization of α-AlH_3 was determined by XRD, SEM and DSC. The XRD results indicate that the product is α-AlH_3. The SEM observation shows that the morphology of α-AlH_3 is mainly lathy. The DSC measurement result shows that the decomposition of α-AlH_3 begin at about 454K.

Key words: α-AlH_3; preparation; characterization
CLC number: TG139$^+$.7 Document code: A Article ID: 0529-6579 (2007) Sl-0117-02

1 Introduction

Aluminum hydride has seven nonsolvated phases. The most stable phase, α-AlH_3, is a possible hydrogen source for automotive fuel cells[1].

However, α-AlH_3 is not currently commercially available. So α-AlH_3 is prepared and the effect of distillation method and distillation temperature on the preparation is discussed in this study. α-AlH_3 was prepared by organometallic synthesis route[2].

2 Experiment

2.1 Materials

All chemicals and solvents obtained from commercial sources were analytical grade. The preparation is extremely sensitive to moisture. So it must be performed in the absence of water. The operation involves two stages: reaction stage and distillation stage. The distillation temperature can be remained the same temperature (one-step). It can also be carried out in two or three steps (two-step or three-step). The reaction is shown below.

$$3LiAlH_4 + AlCl_3 \longrightarrow 4AlH_3 + 3LiCl$$

The X-ray diffraction patterns were acquired with a Philips X′Pert Pro diffractometer using Cu Kα radiation. The morphology was observed with scanning electron microscope (Hitachi S-4800). Thermal analyses were performed with a TG/DSC apparatus (NETZSCH STA 409 PC).

3 Results and discussion

3.1 The effects of distillation method and distillation temperature on the phase

The results of X-ray diffraction are shown in figure 1 and figure 2. All diffraction peaks were in agreement with α-AlH_3 (JCPDS 23—761). This suggests that the product is pure α-AlH_3. It indicates that α-AlH_3 can be successfully produced by different distillation methods and the distillation temperature has a large range. Compared with others[1—4], this method can be operated more easily.

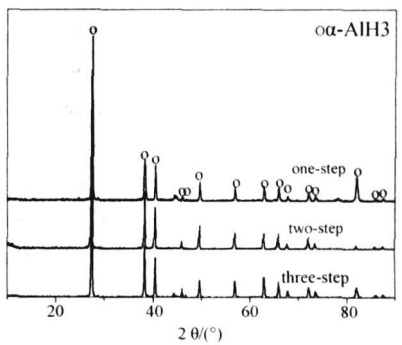

Fig. 1 XRD patterns of α-AlH_3 prepared by different distillation step

①收稿日期: 2007-01-10
基金项目: 国家高技术研究发展 863计划资助项目 (2006010521053)
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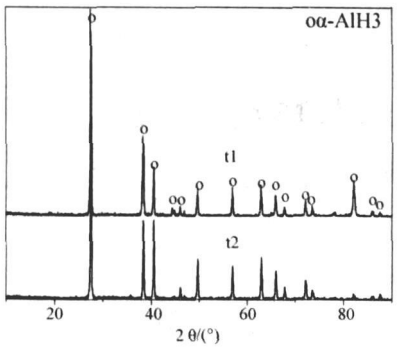

Fig. 2 XRD patterns of α-AlH_3 prepared by different distillation temperature ($t_2 - t_1 = 6$K)

3.2 The effects of distillation temperature on the morphology

The SEM micrographs of α-AlH_3 samples prepared by different distillation temperature respectively are shown in Figure 3. The morphology of α-AlH_3 is mainly lathy. The other morphology is bulk aggregation. In Figure 3, we can find that the ratio of lathy α-AlH_3 increased with increasing distillation temperature. It indicates that the distillation temperature determines the growth rate and the oriented degree of the α-AlH_3 when other conditions keep same.

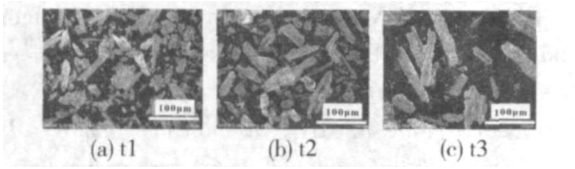

Fig. 3 SEM images of α-AlH_3 prepared by different distillation temperature ($t_1 < t_2 < t_3$)

3.3 Decomposition temperature

The DSC ppt of α-AlH_3 is shown in figure 4.

There is an large endothermic peak is observed around 465K. The initial decomposition temperature is 454K. This value is above than the values measured by other reports[1, 2, 4]. The possible reason is the difference of surface states. There is an oxide film at the surface. The oxide film can delay the decomposition of α-AlH_3. The thickness of oxidized film is a principal factor which influences the initial decomposition temperature[2].

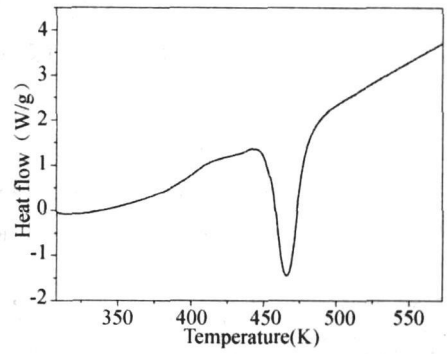

Fig. 4 DSC plot of α-AlH_3 in the temperature range of 308K—573K

4 Conclusion

α-AlH_3 can be successfully produced by one-step, two-step or three-step distillation methods separately with a large distillation temperature range. The morphology of α-AlH_3 are mainly lathy and the ratio of lathy α-AlH_3 increased with increasing distillation temperature. The decomposition of α-AlH_3 begins at about 454K.

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氢化铝的制备与表征

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摘 要: 使用有机金属合成法制备了 α-AlH_3。采用 X射线衍射、扫描电镜和差示扫描量热法对其进行表征。X射线衍射结果表明产品为 α-AlH_3。扫描电镜结果表明, 其形貌主要为长条状。热分析表明其起始分解温度为 454K。

关键词: 氢化铝; 合成; 表征

中图分类号: TG139+.7