Fining Technology of Al-50% Si Powder – A Material for Hermetically-Sealed Casing of Multichip Subsystem

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Abstract

A material for hermetically-sealed casing of multichip subsystem needs the characteristics such as high thermal conductivity, low density, low thermal expansion coefficient, automatic sealing, simplicity and convenience of subsequent application workmanship. The material that is mostly suitable to be used is Al-50% Si compound material prepared by the spray deposition method. The powder metallurgy process (PM) was studied for preparing the Al-Si compound material in this paper. The median particle size of the Al-Si powder must be less than 5μm. The ball-milling technique was adopted to reduce the particle size of Al-Si powder. The result shows: the change in ball-milling speed affects the particle size of Al-50 Si powder greatly and the increase of rotating speed can facilitate powder fining and homogenization. By determining other process parameters, it is found the ball-milling time is the major factor affecting the particle size. As the ball-milling time extends, the particle size reduces. Compared to the dry milling process, the alcohol wet milling process can improve the fine powder output, reduce particle size distribution range, avoid powder agglomeration and get good homogenization. Less particle size also result in higher density and less Si phase particle size in Al-50 Si compound. In the following condition, namely the ratio of grinding media to material is 5:1, the rotating speed 350 rpm and ball-milling time 20 hrs, the median particle size of Al-50 Si powder is 4.64μm.

Keywords: Al-Si compound material; Powder preparation; Ball-milling method

Introduction

As the people keep on pursuing mini-type, light-weight, multifunctional and high-powered electronic equipments, the multichip subsystem (a system-level or a subsystem-level module and assembly in which analog circuit, digital circuit and microwave power circuit are hermetically sealed by adopting the multichip packaging technology) becomes more and more popular. Since the multichip module and assembly needs high package density and some contain the power circuit, there is high requirement on the thermal conductivity of hermetically-sealed casing and power chip’s heat sink. Meanwhile, since there is high requirement on the mini size and light weight, the density of casing is also very sensitive to the electronic package. Besides, the multilayer ceramic substrate or unpacked chips are used when making electronic package and their thermal expansion coefficient is lower, the thermal expansion coefficient of electronic package material must also be lower as much as possible.

By making comparative analysis on the characteristics that the hermetically-sealed casing of multichip subsystem must be available, namely high thermal conductivity, low density, low thermal expansion coefficient, automatic sealing, simplicity and convenience of subsequent application workmanship and reworking property, the material that is mostly suitable to be used is Al-50% Si compound material. Al-50 Si compound material is aluminum (silicon)-based compound material. By changing the content of aluminum and silicon, the aluminum (silicon)-based compound for different uses can be obtained. Now this material has become a very promising electronic package material, especially in the high technology field.

Currently in America, England, China and Japan, the research on high silicon aluminium has made great progress. Upon the different methods, namely fusion casting technology [1-3], powder metallurgic method, spray deposition method [4-8], infiltration process and other billet preparation technologies, as well as the hot extrusion technology, semi-solid extrusion technology, hot forging technology and other molding technology [9], the Al-Si compound material with the Si content between 30% and 70% can be prepared. As for the microscopic structure of the Al-Si compound material prepared by using the fusion casting technology, it has the eutectic silicon phase and looks like the coarse needle flake, as well as the primary silicon phase looks like the large block. The bulky texture characteristics deteriorates the physical property and the processability of material; moreover the increase of silicon content can intensify the problem. As the new technology of near net shape material featured in rapid setting, the spray deposition method developed by Britain Osprey Corporation can prevent aluminium and silicon from getting rejection reaction and avoid from solidification segregation in the moulding process; moreover the spray deposition preparation method allows to make repeated production and leave the content of silicon to be 70% [10].

In this paper the powder metallurgy technology (PM) was used for preparing the Al-Si compound material sample, and the Al-40 Si alloy powder of 300 meshes and the Si powder of 500 meshes were used for raw materials. In order to guarantee the Al-Si compound material to be available good performance, the particle size of the Al-Si powder must be less than 5μm according to references. In such case the least texture structure of material can be obtained. How to prepare fine and homogeneous Al-Si alloy powder is the important key technology of the project. It is very difficult to obtain Al-Si alloy powder with a...
median particle size of less than 5 μm, because the aluminium powder has poor grindability, and is easily oxidized and explosive.

In order to make further exploration on the powder metallurgy technology of the Al-Si compound material with high silicon contents, it is important to discuss the technical feasibility of PM technology, study the influence of the ball-milling process parameters on the particle size of Al-50 Si alloy powder, and so study the influence of particle size on sintering density of Al-Si compound.

**Experimental Procedure**

**Experimental equipments**

Planetary ball mill (QM4SP), Low-oxygen glove box, Laser diffraction particle size analyzer (MASTERSIZE 2000).

**Experimental raw material**

Al-40 Si alloy powder (300 mesh) and Si powder (500 mesh).

**Experimental method**

As for the Al-50 Si powder, it was preferred to adopt the process route of high-energy ball-milling and mixing of (Al-40Si) + Si. In the glove box full of high-purity argon, Al-40Si alloy powder, Si powder and steel ball were put into the ball-milling tank, and then sealed full of Ar gas.

It is necessary to study the influence of ball-milling process parameters, namely the rotating speed (250–350 rpm), time (5–20 hrs) and ratio of grinding media to material (3:1, 5:1) on particle size of Al-50 Si powder, and study the influence of particle size on sintering density of Al-50 Si compound. In the end, the best milling process parameter could be obtained accordingly.

**Results and Discussion**

**Results**

After changing the rotating speed, time, ratio of grinding media to material and other parameters, the particle size of Al-50 Si powder to be measured is detailed in Table 1 and 2 and Figures 1-5.

**Discussion**

1) By comparing A-11 and A-13, it is found that the change in ball-milling speed affects the particle size of Al-50 Si powder greatly. Increasing the rotating speed equals to enlarge the energy carried by the ball mill, increase the impact force and frequency; in this case the refinement and homogenization of powder will be expedited and the convection, shear and diffusion of powder will increase in varying degrees. When the rotating speed reaches 350 rpm, the median particle size ($d_{50}$) of powder will reduce by 2 μm almost.

2) By comparing A-12 and A-13, it is found the way to increase the ratio of grinding media to material affects the size of Al-50Si powder slightly; however such impact is not as obvious as that of increasing the rotating speed of ball mill.

3) The batch number from A-13 to A-16, focusing on studying the influence of ball-milling time on Al-50 Si powder size (Figure 1), shows that the ball-milling time, after other parameters of Al-50 Si powder are determined, is the main reason affecting the powder size. As the ball-milling time delays, the size of powder becomes smaller and smaller. However the degree of reduction reduces too. This is rightly because that the ball-milling time becomes a secondary cause after the fine powder surface achieves to a limit. By keeping on the ball milling for 20 hrs, the median particle size ($d_{50}$) of Al-50 Si powder can reach 4.64 μm.

4) By comparing A-13 and A-17 (Figures 2 and 4), it is found that the alcohol wet milling technology can bring the powder smaller than that of the power obtained by the dry milling technology; and the powder produced by the alcohol milling technology won’t agglomerate and has good homogenization. This is rightly because that the aluminium powder is the metal powder with good plasticity. In the dry milling process, the powder may agglomerate after certain time; at this moment the reaction is only the collision between ball and ball, where the further and continuous ball milling will not refine the size of powder obviously. Meanwhile in the dry milling process, ball mill transfers the huge mechanical force to powder, where powder is further squeezed by force, cold welded, hardened and refined; in this process, the powder with disordered particle size distribution and irregular shape will be obtained. In the wet milling process, since the absolute ethyl alcohol is firstly attached to the concave of powder, the sharp corner of powder can be easily crushed, therefore the powder with comparatively uniform size distribution and average small size can be obtained.

5) Figures 3 and 4 shows the relation of ball-milling time and size of Si powder. It is obvious that the Si powder is easy to be milled by balls. Since Si powder is very fragile, it is easy to be smashed to pieces.

6) When the median particle size of Al-50 Si powder was 14.82, 9.81 and 4.64 μm, the relative density of Al-50 Si compound is 99.1%, 99.4% and 99.6%, respectively, vacuum hot pressed at 550°C, 60MPa.

<table>
<thead>
<tr>
<th>Batch No.</th>
<th>Ratio of ball to material</th>
<th>Ball-milling rotating speed (rpm)</th>
<th>Time (h)</th>
<th>Method</th>
<th>median particle size $d_{50}$ (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A-11</td>
<td>5:01</td>
<td>250</td>
<td>5</td>
<td>Alcohol wet milling</td>
<td>14.82</td>
</tr>
<tr>
<td>A-12</td>
<td>3:01</td>
<td>350</td>
<td>5</td>
<td>Alcohol wet milling</td>
<td>13.89</td>
</tr>
<tr>
<td>A-13</td>
<td>5:01</td>
<td>350</td>
<td>10</td>
<td>Alcohol wet milling</td>
<td>13.19</td>
</tr>
<tr>
<td>A-14</td>
<td>5:01</td>
<td>350</td>
<td>20</td>
<td>Alcohol wet milling</td>
<td>9.81</td>
</tr>
<tr>
<td>A-15</td>
<td>5:01</td>
<td>350</td>
<td>5</td>
<td>Dry milling</td>
<td>6.46</td>
</tr>
<tr>
<td>A-16</td>
<td>5:01</td>
<td>350</td>
<td>5</td>
<td>Dry milling</td>
<td>4.64</td>
</tr>
<tr>
<td>A-17</td>
<td>5:01</td>
<td>350</td>
<td>5</td>
<td>Dry milling</td>
<td>15.1</td>
</tr>
</tbody>
</table>

Table 1: Comparison on Al-50 Si powder particle size at different process parameters.

<table>
<thead>
<tr>
<th>Powder No.</th>
<th>Median particle size(μm)</th>
<th>Sintering density(μg/cm²)</th>
<th>Relative density(%TD)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A-11</td>
<td>14.82</td>
<td>2.48</td>
<td>99.1</td>
</tr>
<tr>
<td>A-14</td>
<td>9.81</td>
<td>2.485</td>
<td>99.4</td>
</tr>
<tr>
<td>A-16</td>
<td>4.64</td>
<td>2.49</td>
<td>99.6</td>
</tr>
</tbody>
</table>

Table 2: Influence of particle size on sintering density of Al-50Si compound hot pressed at 550°C, 60MPa.
Figure 1: Influence of ball-milling time on particle size distribution of Al-50 Si powder.

(a) 5 hrs ($d_{50}=13.19\mu m$)  
(b) 10 hrs ($d_{50}=9.81\mu m$)  
(c) 15 hrs ($d_{50}=6.46\mu m$)  
(d) 20 hrs ($d_{50}=4.64\mu m$)

Figure 2: Influence of wet milling and dry milling on particle size distribution of Al-50 Si powder.

(a) wet ball-milling ($d_{50}=13.19\mu m$)  
(b) dry ball-milling ($d_{50}=15.10\mu m$)
(a) Original Si powder ($d_{50}=38.08 \mu m$)
(b) 4 hrs ($d_{50}=16.09 \mu m$)
(c) 6 hrs ($d_{50}=9.87 \mu m$)
(d) 8 hrs ($d_{50}=3.69 \mu m$)

Figure 3: Influence of ball milling time on particle size distribution of Si powder.

Figure 4: Relation between ball-milling time and particle size.
(Figure 5) were microstructures of Al-50 Si compounds; the Si phase particle size was 5.6, 4.7 and 3.7 μm, respectively.

**Conclusion**

1) The change in ball milling speed affects the size of Al-40 Si powder greatly, and the increase of rotating speed can expedite powder refining and homogenization.

2) After other parameters are determined, ball milling time is the main reason affecting the particle size. As the ball milling time extends, the particle size of powder reduces.

3) The alcohol wet milling technology can bring the powder with the size smaller than that of the powder produced by the dry milling technology, besides the former has smaller powder distribution range, good size homogenization, and won’t agglomerate. Less particle size also result in higher density and less Si phase particle size in Al-50 Si compound.

4) In the following condition, namely the ratio of grinding media to material is 5:1, the rotating speed 350rpm and ball-milling time 20 hrs, the median particle size of Al-50 Si powder obtained is 4.64 μm.

**References**


