Preparation of Al/Si functionally graded materials using ultrasonic separation method

*Zhang Zhongtao\(^1\), Li Tingju\(^{1,2}\), Yue Hongyun\(^1\), Zhang Jian\(^1\), Li Jie\(^1\)

(1. School of Materials Science and Engineering, Dalian University of Technology, Dalian 116023, China; 2. State Key Laboratory of Materials Modification by Laser, Ion and Electron Beams, Dalian University of Technology, Dalian 116023, China)

Abstract: Functionally graded materials (FGM) have been widely used in many industries such as aerospace, energy and electronics. In this experimental study of fabricating FGM, an approach was developed to prepare Al/Si FGM using power ultrasonic separation method. Material sample with continuously changing composition and performance/properties was successfully produced. Results showed that the microstructure of the FGM sample transited, from its top to bottom, from the hypereutectic structure with a large quantity of primary Si gradually to the eutectic, and finally to the hypoeutectic with numerous primary Al dendrites. The distribution of primary Si and microhardness of the FGM sample also presented graded characteristics, resulting that the wear resistance of the FGM sample decreased from top to bottom. Preliminary discussion was made on the mechanism of the formation of Al/Si FGM.

Key words: functionally graded materials (FGM); aluminum alloys; ultrasonic cavitation; acoustic streaming; degassing

CLC number: TG146.2

Functionally graded materials (FGM) have been developing rapidly in the past two decades, which varies continuously in composition and/or microstructure. Because FGM facilitate the evolution of mechanical, physical and chemical properties of materials\(^{[1-3]}\), they have been widely used in aerospace, energy, electronics and other industries. With the increasing attention of researchers, a series of methods have been used in the preparation of FGM including powder metallurgy, vapor deposition, self-propagation, centrifugal casting and magnetic separation\(^{[4-8]}\). All the methods above have their own drawbacks, however, such as high cost and complex technique.

As a relatively new technique with the advantages of environmental friendly, low cost and equipment simplicity, power ultrasonic field has been widely used in microstructure evolution, light alloy degassing and purification\(^{[9-13]}\). Some researchers have studied the movement behaviors of suspended particles in solutions under ultrasonic field\(^{[14-16]}\). Considering that the nonmetallic particles in molten melt can also migrate upwards under ultrasonic field, the idea to prepare FGM using ultrasonic separation method has been proposed in this research. By now, no reference papers have reported the preparation of FGM by means of ultrasonic field. In this study, a novel method was developed to prepare Al/Si FGM using ultrasonic separation of primary Si particles. The structure, primary Si distribution, microhardness and wear resistance of the material samples were investigated in detail. In addition, the mechanism of this Al/Si FGM process was preliminarily discussed.

1 Experiment

1.1 Experimental apparatus

Al-18wt%Si hypereutectic alloy with the liquidus temperature of 657°C and the solidus temperature of 577°C was used in the experiment. The experimental installation consisted of an electrical resistance furnace, a temperature recording system and an air-cooled ultrasonic system. The ultrasonic system was composed of a 20 kHz ultrasonic generator, a Lead-Zirconate-Titanate (PZT) piezoceramics transducer, and a cylindrical radiator bar which was made of Ti-6Al-4V alloy and 25 mm in diameter. The output power of the ultrasonic apparatus was adjustable with the maximum of 1.5 kW.

1.2 Experimental procedure

The prepared Al-18wt%Si alloy was melted in the electric resistance furnace to 750°C, and held for 30 min to make Si element distributed uniformly in the whole melt. The preheated (ultrasonic) radiator bar was started first, and then, the melt was poured into a preheated cylindrical graphite crucible of 50 mm diameter designed with a taper for sample removal. The radiator bar was conveyed from the top of the melt and the depth of immersion was 10 mm. Type-K thermocouple
and self-developed temperature analysis system were used to measure and record the temperature of the melt. The beginning temperature of ultrasonic vibration was set at 700℃. When the temperature was below 585℃, the viscosity was too high for primary Si to migrate. Therefore, ultrasonic vibration was stopped at 585℃ and the bar was pulled out. The applied power was 300 W and effective action time was nearly 180 s with the average cooling rate at 0.4 °C/s. The sample was cooled in atmosphere to room temperature, and Al/Si FGM sample was obtained. The original samples were made as a contrast without ultrasonic vibration or other degassing treatment in the entire preparing processes.

The samples were cut longitudinally in the middle, mechanically polished and lightly etched with 1%HF-water reagent. The microstructure of the samples was characterized by using of MEF4 optical microscope and JSM-5600 LV SEM. The microhardness was measured with a HVS-5 Vickers hardness tester. The wear resistance of the samples was tested on a UMT-2 multifunctional friction and wear tester. The wear test parameters were as follows: loading, 5 N; wearing time, 10 min; travel, 8 mm; velocity, 2 mm/s; grinding ball, carborundum ball with 4.7625 mm in diameter. To minimize random errors, a series of representative metallographs in the longitudinal direction were analyzed using Image-Pro Plus (IPP) to measure the average volume fraction of primary Si particles.

2 Results and discussions

Figure 1 shows the macrographs of the samples with and without ultrasonic field. The micrograph selected randomly from the original sample is shown in Fig. 2(a). The size of primary Si in the original sample was less than 100 µm. In region A and the upper region B of the Al/Si FGM sample, the microstructure was Al-Si hypereutectic structure with a great number of primary Si. The primary Si size was mostly in the range from 130 µm to 200 µm, as shown in Fig. 2(b), (c). It can be seen in Fig. 2(d) that the microstructure changed from hypereutectic structure with a number of primary Si to eutectic structure, and the primary Si size varied from 60 µm to 130 µm in the transition area. In the lower part of the sample, shown in Fig. 2(e), the microstructure was hypoeutectic structure with a great number of primary Al dendrites and almost no primary Si phase existed there. In a word, the microstructure of the obtained FGM sample varied from hypereutectic to eutectic and then to hypoeutectic from top to bottom of the sample; and the size of primary Si also decreased from top to bottom at the meantime, due to the upward migration of primary Si particles under power ultrasonic field.

In the Al-18wt%Si alloy, the primary Si particles are non-wettable by Al melt. These particles tend to adsorb gaseous phases on their surface and at surface defects, so they can easily be the potential nuclei of cavitation bubbles. During the propagation of ultrasonic field in melt, it generates alternating expanding and compressing pressure fluctuation. If the pressure exceeds the cavitation threshold, numerous of cavitation bubbles can be formed around primary Si particles. Most of the cavitation bubbles absorb gases such as hydrogen and oxygen inside, and grow into larger bubbles, due to the difference in the interface area \(^{[17]}\). The whole process proceeds very fast owing to ultrasonic cavitation. When the bubbles are big enough to overcome their drag forces and gravities, they can float to the melt surface. In the course of float, the cavitation bubbles can catch the primary Si particles with them, leading to the upward migration of primary Si particles. The mechanism of such process is schematically shown in Fig. 3 \(^{[18]}\).

The viscosity of melt increases with the fall of melt temperature. In the latter period of solidification, the viscosity was so high that the primary Si particles could not migrate anymore, and they settled down with graded content distribution and thus formed Al/Si FGM. Acoustic streaming...
also played a role in the migration of Si particles, such nonlinear convection flow could help to transport bubbles and primary Si particles upwards and thus enhance the formation of Al/Si FGM.

From the metallographs of the Al/Si FGM sample, it was found that the size of primary Si particles increased in accordance with the content increase from region C to region A, from less than 100 µm to mostly 130 µm–150 µm and to nearly 200 µm, as a result of the upward migration of primary Si particles.

Generally speaking, the formation process of Al/Si FGM using ultrasonic separation method could be divided into four stages: (1) nucleation of cavitation bubbles around primary Si particles, (2) formation of large bubbles under ultrasonic field, (3) floatation of primary Si particles along with bubbles, (4) dwelling of primary Si particles under high viscosity and formation of Al/Si FGM.

The particle volume fraction vs distance from top of the Al/Si FGM sample was measured using IPP, as shown in Fig. 4. In the sample with ultrasonic field, the content of primary Si decreased gradually from top to bottom with the maximum content at 18.3% in the top area. Almost no primary Si particle existed in region C. The content of primary Si in upper portion was higher than the 6.7wt% average content in the original sample, and lower than that average content in the bottom portion. The primary Si particles, however, distributed homogeneously in the sample that without ultrasonic field.

The results of particle volume fraction distribution agreed well with the changes in Figs. 1 and 2. Because of no heat preservation above the crucible, the solidification rate was higher in the upper, leading to a fluctuation in primary Si content. With proper modification of the existing equipment, sample with better graded distribution will be obtained.

The microhardness distribution of the samples in the longitudinal direction is shown in Fig. 5. The hardness of Al/Si FGM decreased gradually from region A to region C, corresponding well to the changes of microstructure and primary Si content distribution. Primary Si is a strengthening phase in Al/Si alloy, and therefore, the hardness was mainly determined by the content of primary Si. The hardness increased from bottom to top, due to the upward migration of primary Si with ultrasonic field. Meanwhile, the hardness was also affected by porosity in the sample. Ultrasonic field could significantly remove the porosity and thus increase hardness. The microhardness distribution agreed well with the distribution of primary Si and porosity.

The wear resistance of region A, B and C of the FGM sample was evaluated. The morphologies of the grinding cracks (or scars) are shown in Fig. 6. As long as the width of the grinding crack is measured, the volume of the crack can
be calculated. The wear resistance performance of the FGM sample is characterized by the volume of the grinding crack, that is, the greater the volume, the worse the wear resistance of the sample. The width $Y$ of grinding crack in region A was at 350 $\mu$m, much smaller than those in regions B and C, at 652 $\mu$m and 740 $\mu$m, respectively. It can be seen that the morphology of the grinding crack in Fig. 6(a) was more in order comparing to those in Fig. 6 (b) and Fig. 6(c), and that the resulted volume loss was also greater than the other two.

Since the primary Si is much higher in hardness than Al and it serves as a strengthening phase to the Al substrate, the wear resistance performance of the sample is mainly affected by the distribution of primary Si. Under power ultrasonic field, primary Si particles in the FGM sample migrated upwards, causing the variation in wear resistance from top to bottom of the sample. The results of the wear resistance test are given in Table 1. The wear resistance performance of the FGM sample has the same distribution as the primary Si and hardness. The wear resistance of the upper region is about nine times as much as that of the bottom of the FGM sample, which can be utilized for the fabrication of functional materials such as for gears, etc.

![Fig. 5 Microhardness distribution of the samples](image)

![Fig. 6 The morphologies of the grinding cracks of Al/Si FGM sample: region A (a), region B (b), region C(c)](image)

<table>
<thead>
<tr>
<th>Table 1 Wear test results of the FGM sample</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample</td>
</tr>
<tr>
<td>---------</td>
</tr>
<tr>
<td>Region A</td>
</tr>
<tr>
<td>Region B</td>
</tr>
<tr>
<td>Region C</td>
</tr>
</tbody>
</table>

The mechanism of Al/Si FGM preparation using ultrasonic field is still not clear at the moment. Further research on mechanism, experiment apparatus and parameter control will be conducted in future work to produce more ideal FGM.

3 Conclusions

(1) A novel approach for the preparation of Al/Si FGM was successfully developed using power ultrasonic separation method, which has not yet been reported by other researchers.

(2) The microstructure of the obtained FGM changed from Al-Si hypereutectic with a large number of primary Si, then to eutectic, and finally to hypo-eutectic with a large number of primary Al dendrites in the longitudinal direction.

(3) The volume fraction distribution, microhardness variation and wear resistance of the FGM sample presented well graded characteristics, corresponding well to the microstructure distribution of the Al/Si FGM.

References


This work was financially supported by the National Natural Science Foundation of China (No.50474055).