Microstructural Characteristics and Vibration Fracture Properties of Al-Mg-Si Alloys with Excess Cu and Ni

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This study investigated the effect of co-addition of Cu and Ni on the microstructure and vibration properties of Al-Mg-Si alloys. The results show that additions of Cu and Ni resulted in the formation of Al3(Ni,Cu) phase, a harder Al matrix and an accelerated aging rate. Nanoindentation analysis indicates that the Al3(Ni,Cu) phase exhibits a hardness of 7.7 GPa and Young’s modulus of 141.2 GPa. Due to the increased modulus by reinforcement effect of Al3(Ni,Cu), the alloyed samples possessed a higher resonant frequency. However, the damping capacity was reduced with alloying because of the hardened matrix. It was also found that since microcracks could be generated from the irregular-shaped Al3(Ni,Cu) in the vicinity of the tip of the main crack when suffering vibration deformation, the crack propagation resistance of the alloyed samples was inferior. [doi:10.2320/matertrans.48.854]

(Received December 15, 2006; Accepted February 19, 2007; Published March 25, 2007)

Keywords: aluminum-magnesium-silicon, resonant vibration, damping capacity, nanoindentation

1. Introduction

Al-Mg-Si wrought alloys have been widely used in transportation systems owing to their fair strength, weldability and corrosion resistance. The precipitation sequence of solution-treated 6xxx alloys as represented by the Al-Mg-Si ternary during artificial aging can be reported to be: a supersaturated solid solution (SSS) → GP-I zones → metastable needle-like β′ precipitates (or called GP-II zones; formed through the transformation of GP-I as nuclei) → metastable rod-like (or lath-like) β precipitates → stable β phase.1

In the application of structural materials in dynamic systems, undesirable effects resulting from mechanical vibration, such as noise, fatigue or even failure of components, should be taken into consideration.2 In the past decade, several research works have been devoted to the vibration behavior of Al-Mg-Si alloys.3–6 With respect to the effect of aging conditions, it was found that aging conditions significantly affect the resonant vibration properties, such as vibration deformation feature and damping capacity. The peak aged samples possess a poor damping capacity mainly due to the decrease in dislocation mobility.6

Among the elements added to 6xxx series alloys for increasing strength and grain-size control, copper has arrested considerable attention. Cu additions reduce the natural aging rate of Al-Mg-Si alloys but generally increase the kinetics of precipitation during artificial aging.7 In addition to the phases that precipitate in the ternary alloys, the equilibrium precipitate in high Cu Al-Mg-Si-Cu alloys, Q, has been identified as Al5Cu2Mg5Si6 and Al5CuMg5Si6.8,9

Additions of Ni lead to the formation of Al3Ni in the aluminum matrix through eutectic reaction during solidification. Due to the high Young’s modulus and tensile strength (~2160 MPa), the compound Al3Ni has been applied as the reinforcement for aluminum-matrix composites.10,11 A report regarding the properties of the Al-Zn-Mg/Al3Ni system indicates that an increased amount of Al3Ni reinforcement not only raises the yield strength but also reduces the time for peak aging.12

Considering Cu and Ni could strengthen aluminum alloys through precipitation and dispersion hardening respectively, the objective of this study is to explore their influence on the resonant vibration behavior of an Al-Mg-Si alloy. The relationships between microstructure, damping capacity and vibration fracture resistance will also be clarified.

2. Experimental Procedures

The test materials of which the compositions are listed in Table 1 are typical 6061 and 60X with the Cu content of 0.81 mass% and the Ni content of 1.41 mass%. Extruded 6061 and 60X sheets were solution treated at 540°C in an air furnace using deoxidation coating for 2 hours and then water quenched. The artificial aging treatment was performed at 170°C in an oil bath. Microstructural observation on the samples was carried out with optical microscope (OM) and transmission electron microscope (TEM).

In order to collect mechanical data for reference, Rockwell hardness test was performed to obtain the hardness change during aging process. The mechanical performance of individual phase in the peak aged samples was evaluated by nanoindentation testing using a MTS Nano indenter XP. A continuous stiffness measurement (CSM) technique was used during indentation. A purely isotropic silica standard was used to calibrate the system, and a pure Al sample was also prepared for comparison. The indentation experiment used in this study can be summarized as follows: (1) the surface position is determined by approaching the indenter to the

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Table 1 Chemical composition of the alloys (mass%).

<table>
<thead>
<tr>
<th>sample/element</th>
<th>Mg</th>
<th>Si</th>
<th>Cu</th>
<th>Fe</th>
<th>Mn</th>
<th>Cr</th>
<th>Ni</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>6061</td>
<td>1.12</td>
<td>0.73</td>
<td>0.30</td>
<td>0.13</td>
<td>0.11</td>
<td>0.09</td>
<td>—</td>
<td>Bal.</td>
</tr>
<tr>
<td>60X</td>
<td>0.92</td>
<td>0.71</td>
<td>0.81</td>
<td>0.17</td>
<td>0.37</td>
<td>0.09</td>
<td>1.41</td>
<td>1.41</td>
</tr>
</tbody>
</table>

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sample at a 10 nm/s rate; (2) after surface contact, the indenter is loaded under displacement control, i.e., at a loading rate such that its displacement rate is 100 nm/s or 1 nm/s, and the loading continues until a total indenter displacement is reached (2000 nm for Al matrix and 500 nm for Al\textsubscript{3}(Ni,Cu)); (3) The load-displacement data obtained were analyzed using the method of Oliver and Pharr\textsuperscript{13} to determine the hardness and the elastic modulus as a function of the displacement of the indenter. Each datum was the average of at least five tests.

A simple cantilever beam vibration system was used for the vibration experiments and damping measurements (Fig. 1(a)). The test specimen, Figure 1(b), rectangular with dimensions 15 mm × 80 mm × 3 mm was mounted and fixed on end to the vibration shaker. Two V-notches near the clamp were made to restrict crack initiation from the notch front.

The vibration force was monitored using an acceleration sensor, and the deflection amplitude of the specimen at the end opposite the vibration shaker was detected by a deflection sensor once every 10 seconds. The resonant vibration tests were conducted at either a fixed vibration force (1.5 G, where G denotes the acceleration due to gravity, 9.8 m/s\textsuperscript{2}) or a fixed initial deflection (ID) under resonance. The resonance frequency was taken as the frequency leading to the largest deflection and determined by varying the vibration frequency continuously. The variation in deflection amplitude against the number of cycles was recorded. Each datum was the average of results from more than 3 samples.

Damping capacity was measured in terms of logarithmic decrement (δ value) which was derived from the deflection amplitude decay of a specimen under free vibration. Logarithmic decrement value is defined as follows\textsuperscript{14}

\[
\delta = \frac{1}{n} \ln(A_i/A_{i+n})
\]

where \(A_i\) and \(A_{i+n}\) are the deflection amplitude of the \(i\)th cycle and the \((i + n)\)th cycle separated by \(n\) periods of oscillation.

3. Results and Discussion

3.1 Microstructural observation and hardness tests

Figure 2 shows the microstructure of the solution treated samples. The matrix of the 6061 specimens shows an equiaxed grain structure. Numerous intermetallic particles with the size of several microns and the area fraction of about 8.3% could be observed in the 60X samples. The TEM results, diffraction patterns and EDS data shown in Fig. 3, demonstrate that the intermetallic compound (IMC) exhibited an orthorhombic Al\textsubscript{3}Ni structure. A certain amount of solute was found dissolved in it, mainly Cu. Thus, this intermetallic phase can be identified as Al\textsubscript{3}(Ni,Cu).

Figure 4 illustrates the Rockwell hardness of the samples against the aging time. It could be found that the 60X was harder than 6061 during the whole aging process even under the solution treated condition. For each test material, the hardness increased rapidly at the early stage (within the first 6 hours) of artificial aging. The hardness of the 6061 sample reached a maximum value (65.4 HRB) after being aged for 20 hours, while the 60X sample spent a shorter aging time (16 hours) to achieve a greater peak hardness (75.6 HRB).

Figure 5 shows the representative load-displacement curves for the Al matrices and Al\textsubscript{3}(Ni,Cu) particles of the
peak-aged samples. To avoid the matrix effect, the depth for the indentation was set to be 500 nm for Al₃(Ni,Cu) and that for the Al matrices was 2000 nm. It can be observed that with an identical load, 20 mN, the penetration of the indenter into Al₃(Ni,Cu) was less deeper than that into the Al matrices. The matrix of 60X seemed to have a slightly greater deformation resistance than that of 6061. Table 2 displays the hardness and Young’s modulus results for testing of various phases in the discussed depth ranges. It indicates that the Al₃(Ni,Cu) phase exhibited an average hardness of 7.7 GPa and Young’s modulus of 141.2 GPa, which were much greater than those for the matrices. The modulus measured in this study was within the reported modulus range of Al₃Ni (116 GPa~152 GPa). This implies that dissolution of 2.3 at%Cu doesn’t affect the mechanical behavior of Al₃Ni too much. The indentation test of the Al matrices was performed under different strain rates, 100 nm/s and 1 nm/s. The results in Table 2 indicate that the strain rate affected the hardness and modulus measured. This may account for the higher value of the modulus compared to those in literature[15] and needs further investigations to prove. The influence of Cu and Ni co-additions on the elastic modulus of the Al matrix was very limited (see data of 60X and 6061). However, the hardness for the matrix of the 60X was greater than those of 6061 to a detectable extent under both the testing conditions. Considering the performance of the bulk material, it could be
deduced that the 60X sample possess greater hardness and modulus, if the rule of mixture is applied.

3.2 Vibration properties and vibration fracture resistance

The resonant frequencies of the 6061 and 60X specimens are 88 Hz and 93 Hz respectively. McGuire et al. reported that the resonant frequency of materials depends on the effective elastic modulus, density and dimensions of the specimen.16,17) As discussed above, the 60X samples had a higher modulus than 6061. Thus, the raised resonant frequency of 60X might be attributed to the reinforcement effect by Al$_3$(Ni,Cu).

Conducting the vibration test at the resonant frequency of each sample, the D-N curves (deflection amplitude vs. number of vibration cycles) under a constant push force, 1.5 G, are illustrated in Fig. 6(a). The D-N curves can be divided into a short initial stage (Stage I) with ascending deflection amplitude, a second stage in which deflection remained constant (Stage II), and a final stage with a descending deflection amplitude (Stage III). The ascending and constant deflection amplitudes within Stages I and II can be attributed to the effect of strain hardening in competition with that of crack generation and linking within this region.18) The descending deflection in Stage III is due to the deviation of the actual vibration frequency from the resonant frequency caused by the inward propagation of major cracks. In this study, vibration life is defined as the number of vibration cycles at the beginning of Stage III.

The initial deflection is inversely proportional to the damping capacity.6) That is, a sample with a lower initial deflection exhibits a superior ability to dissipate vibration energy. Based on the data shown in Fig. 6(b), the initial deflection of the 60X samples was about 3.0 mm, apparently greater than that of 6061 (about 2.8 mm). This corresponds with the damping capacity data shown in Table 3. On account of the higher damping capacity and thus reduced vibration deformation, the critical number of cycles to failure was $8.3 \times 10^4$ for 6061, which was obviously greater than that the 60X, $4.0 \times 10^4$, when the push force was fixed at 1.5 G.

In order to remove the effect of damping capacity, the initial deflection of the 60X sample was reduced to about 2.8 mm by lowering the vibration force to 1.4 G (Figure 6(c)). It was found that the deflection amplitude of the 60X sample still fell faster than 6061. This represents that the 60X specimens still exhibited an inferior vibration fracture resistance under an identical initial deflection condition.

Previous report19) showed that hard particle-reinforced Al alloys can significantly increase the damping capacity due to the internal friction between the dispersoids and the matrix. In this study, Al$_3$(Ni,Cu) of about 8% in area fraction was dispersed in the Al matrix, however, the damping capacity was lower than unalloyed samples. This can probably be ascribed to the matrix hardening effect of the 60X samples which overcome the positive contribution of the intermetallic

<table>
<thead>
<tr>
<th>Indentation rate()</th>
<th>60X-Al$_3$(Ni,Cu)</th>
<th>60X-Al matrix</th>
<th>6061-Al matrix</th>
<th>Pure Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>(\text{H (GPa)})</td>
<td>(\text{E (GPa)})</td>
<td>(\text{H (GPa)})</td>
<td>(\text{E (GPa)})</td>
<td>(\text{H (GPa)})</td>
</tr>
<tr>
<td>100 nm/s</td>
<td>7.735</td>
<td>141.20</td>
<td>1.854</td>
<td>94.001</td>
</tr>
<tr>
<td></td>
<td>±0.460</td>
<td>±9.797</td>
<td>±0.028</td>
<td>±0.729</td>
</tr>
<tr>
<td>1 nm/s</td>
<td>—</td>
<td>—</td>
<td>1.741</td>
<td>80.215</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>±0.099</td>
<td>±1.916</td>
</tr>
</tbody>
</table>
Fig. 6 Vibration test results (a) D-N curves of the samples under a fixed vibration force of 1.5 G and (b) initial deflection amplitude and vibration life estimated from (a). (c) D-N curves under a fixed initial deflection of about 2.8 mm.

Table 3 Vibration properties of the specimens.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Resonant frequency (Hz)</th>
<th>Logarithmic decrement (δ)</th>
</tr>
</thead>
<tbody>
<tr>
<td>6061</td>
<td>88 ± 1</td>
<td>$8 \times 10^{-3}$</td>
</tr>
<tr>
<td>60X</td>
<td>93 ± 1</td>
<td>$7 \times 10^{-3}$</td>
</tr>
</tbody>
</table>

Fig. 7 Crack propagation morphology: (a) 6061, and (b) (c) 60X.
dispersoids to damping capacity, since a hardened matrix due to precipitation causes a decrease in dislocation mobility and thus a lower ability to dissipate vibration energy.6) The inferior vibration fracture resistance (vibration life) of the 60X under either constant force or constant ID conditions could be explained by the crack initiation and propagation feature displayed in Fig. 7. Since the gliding mode of mobile dislocations under peak-aged conditions was supposed to be precipitate bypassing (so-called Orowan looping), no persistent slip band was found.8) With respect to the 60X, the main crack tended to grow through the \( \text{Al}_3(\text{Ni,Cu}) \) particles and led to a more tortuous cracking than 6061. Figure 8(a) illustrates that large side cracks could be found parallel to the main crack. Detailed observation, Figure 8(b), shows that in the vicinity of the main crack tip, a microcrack was found generated from the \( \text{Al}_3(\text{Ni,Cu}) \) particles with an irregular shape. Such microcracks might result in side cracks with the growth direction parallel to the main crack and probably accelerate fracturing. This means in addition to a lack of the ability to blunt the crack growth \( \text{Al}_3(\text{Ni,Cu}) \) may act as a crack nucleation site for side cracks due to the stress concentration around the main crack tip.

4. Conclusions

Co-additions of Cu and Ni into Al-Mg-Si alloys resulted in accelerated artificial aging rate, increased the peak hardness, higher resonant frequency and reduced damping capacity. Orthorhombic \( \text{Al}_3(\text{Ni,Cu}) \) particles with high hardness (7.7 GPa) and modulus (141.2 GPa) were distributed uniformly in the matrix. Microcracks were observed initiated from \( \text{Al}_3(\text{Ni,Cu}) \) around the main crack tip probably due to stress concentration. It might result in the formation of side cracks, the consequent fast crack propagation and thus inferior vibration fracture resistance under the vibration testing conditions in this study.

Acknowledgements

This work has been supported by National Science Council of R.O.C. (Contract: NSC 94-2216-E-259-003), for which the authors are grateful.

REFERENCES