High Temperature Tensile Deformation Behavior of New Heat Resistant Aluminum Alloy

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This study attempted to investigate the high temperature tensile deformation behaviour of new Al-1%Mg-1.1%Si-0.8%CoNi heat resistant aluminum alloy. New aluminium alloy strengthened by Co-Ni based phase was manufactured by using powder ball milling and continuous casting, based on the alloy design & preliminary test. High temperature tensile tests were conducted at various temperatures from 298 K to 723 K. OM, SEM, EPMA, XRD, FIB and HR-TEM equipments were used to analyse microstructure, phases, and fracture surface. Microstructure of the new alloy mainly consisted of Al matrix and Co-Ni based phases (1–8 µm). The Co-Ni based phase was analyzed and confirmed as (Ni, Co)Al₄ having incoherent interface with matrix. In high temperature tensile results, the new aluminum alloy didn’t show significant decrease (19.6%) of strength with increasing temperature (723 K), suggesting totally different behaviour vis-a-vis conventional A319 alloy (87.0% decrease of T.S.). Fractography observation results of new alloy represented the ductile fracture mode with dimples. Voids were mainly initiated at Co-Ni based strengthening phases. It was apparently observed that the strengthening effect of the (Ni, Co)Al₄ could be still maintained at high temperature of 723 K. The deformation mechanism of this alloy was also discussed. [doi:10.2320/matertrans.M2011025]

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1. Introduction

Recently, aluminum alloy has been re-focused as a lightweight material for automobiles, because of its superiority in strength to weight, mechanical properties, formability and recyclable merit. Heat-resistant Al alloy has been used for engine blocks, cylinder heads, and cylinder liners. Much development and many studies are still ongoing for the purpose of improving heat-resistant stability, high-temperature mechanical and fatigue properties.¹ Currently, the most commonly used heat-resistant Al alloy includes A319 and A356.²-⁴

Precipitation of intermetallic compounds by adding a variety of elements is generally used to improve the mechanical properties of Al alloys. However, it also has some limit because the intrinsic characteristic of intermetallic compounds can reduce the properties at high temperatures. For example, in the case of Al-Si-Mg-Fe alloy, brittle intermetallic phases including the precipitates β-(Al₃FeSi) and α-(Al₁FeSi) are unstable at over 473 K. As a result, it can drop the high temperature properties of the alloy.⁵-⁷ Apart from the strengthening by using intermetallic precipitates, studies on Al based composite materials have been also performed by using dispersion particles including SiC, Al₂O₃ and SiO etc.⁸,⁹ to enhance the heat resistance of Al alloy. These Al alloys that are manufactured through the addition of dispersion strengthening particles are relatively superior at high temperatures, but also have some problems like high cost compared to conventional materials, reduced physical properties due to the incoherent interface between dispersion particle and matrix, residual thermal stress caused by the difference of thermal expansion coefficient, and local deviation of properties.⁹ And then, other attempts have been made to improve high temperature resistance using DAS (dendrite arm spacing) control, rapid solidification, and thermo-mechanical treatment. However, the complicate processing controls and high costs still limit the commercialization of those approaches. Accordingly, other attempts to enhance high temperature properties are necessary using new reinforcing phases that are differentiated from conventional methods. Given this need, the authors could confirm the possibility of Co-Ni based reinforcing phases by the examination of basic alloy and the design of alloy composition.

This study, based on the Al-Mg-Si composition, manufactured a new heat-resistant Al alloy that was reinforced with Co-Ni based 2nd phase. The high temperature tensile behavior of this alloy was investigated and the micro-mechanism of deformation was also discussed.

2. Experimental Methods

Al-Mg-Si-0.8%CoNi heat-resistant Al alloy was manufactured using powder ball milling, high-frequency induction melting and continuous casting. Firstly, pure Co and pure Ni powders were mixed and prepared by boll milling process for one hour. These powders were added to the pure Al melts to manufacture the Al-3%CoNi master alloy. This master alloy was re-melted, and Al, Mg, and Si were added to adjust Al-1%Mg-1.1%Si-0.8%CoNi composition. It was directly manufactured into billets by continuous casting. And then, additional heat treatments (783 K/2 h, and 463 K/8 h) were conducted. The composition of the manufactured alloy was identified using ICP analysis as shown in Table 1. SEM was used for microstructural observation and the distribution and sizes of pores and reinforcing phases were measured via

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image analysis. Phase analysis was conducted using XRD, EDS, and EPMA. For more precise analysis, a FIB (Focused ion beam) was used to collect parts containing reinforcing particles. Then, the particles were observed using a HR-TEM, followed by analysis of the diffraction pattern. The billet’s hardness was measured 10 times with a Vickers hardness tester and the average hardness value was 88.9 Hv. From the billet, a specimen of 4 mm in diameter and 12.5 mm in gauge width was machined for a high temperature tensile test. For the high temperature tensile test, an Instron 8501 with a high temperature furnace was used. Here, the test temperatures were 298 K, 403 K, 523 K, 623 K and 723 K. Heating-up speed was 10 K per minute. Each temperature was maintained for 10 min. And then, a high temperature tensile test was performed at a cross head speed of 1 mm/min. After the test, SEM was used to observe fracture surfaces.

3. Results and Discussion

3.1 Microstructural observation and phase analysis results
The results of microscopic observation (un-etched) of the new Al-Mg-Si-CoNi alloy are indicated in Fig. 1(a), (b). Some white particles and pores were observed in the matrix. The white particles were analyzed with an image analyzer and found to have size range of 1~8µm, and an average inter-particle distance of 17.4µm. Phase fraction of white particle was calculated as 2.84%. The size range of pore was 1~7.5µm and the fraction was 1.21%. Optical microstructure for etched specimen is also suggested in Fig. 1(c), representing large grains over 100µm.

Al, Mg, Si, Co and Ni elemental EPMA analysis was performed on the sectional specimen. The results are shown in Fig. 2 and indicate that Al was distributed all over the matrix. Co and Ni were converged into white particles, so this phase was interpreted as the Co-Ni based 2nd phase. Mg and Si elements were locally detected in the pores (black area on the BSE image), possibly due to the segregation in the process of continuous casting.

XRD analysis results are shown in Fig. 3. For this alloy, Al (JCPDS #01-1176) and (Ni, Co)₃Al₄ (JCPDS #44-0922) phases were observed, with some detection of Si (JCPDS #41-1111) phase. For more precise analysis of white particle, FIB was used (after the confirmation of elemental analysis) to manufacture HR-TEM specimen that included the particle. HR-TEM observation results are shown in Fig. 4. The Co-Ni based particle (a) was observed to have an incoherent interface (b) with the matrix Al. However, the particle-matrix interfacial zone appeared to be defect free, exhibiting a perfect metallurgical interfacial bonding. Subsequently, in the result of analysis on the TEM diffraction pattern (Fig. 5), it was re-confirmed that the Co-Ni based phase was (Ni, Co)₃Al₄.

3.2 High temperature deformation behavior
Figure 6 presents the results of the tensile test conducted at different temperatures from room temperature up to 723 K. The tensile strength and elongation at room temperature were 255.4 MPa and 15.7%, respectively. At 723 K, the tensile strength was 205.4 MPa, a 19.6% drop compared to the room temperature, and the elongation was 20.0%, which is a 26.9% increase. To compare the high temperature tensile properties of the new heat-resistant Al alloy used in this study, documentary survey was made for the high temperature tensile results of A319 alloy — one of the most common heat resistant Al alloy. The comparison results are shown in Fig. 7. At over 473 K, high temperature tensile strength of A319 alloy dropped drastically and there was an increase in

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Al</th>
<th>Mg</th>
<th>Si</th>
<th>Co</th>
<th>Ni</th>
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<tbody>
<tr>
<td>Composition</td>
<td>Bal</td>
<td>0.96</td>
<td>0.83</td>
<td>0.36</td>
<td>0.36</td>
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Table 1 Chemical composition of Al-Mg-Si-CoNi alloy used in this study (mass%).
elongation. In contrast, the new heat-resistant Al alloy lost only approximately 50 MPa in tensile strength as the temperature rose from room temperature to 723 K, with a slight increase of elongation. These results confirm that the newly developed heat-resistant Al alloy is superior to conventional A319 alloy in the high temperature tensile properties.

Figure 8 presents the results of fractography on the tensile fractured samples that were tested at room temperature and 623 K (as a typical example of high temperature). The fracture surfaces at both temperatures represented the typical ductile fracture mode containing well developed dimple voids. Most dimples were initiated and developed around
the reinforcing phase, and inside the dimples, remaining \((\text{Ni}, \text{Co})_3\text{Al}_4\) reinforcing particles were observed. The reinforcing particles \(((\text{Ni}, \text{Co})_3\text{Al}_4)\) inside the dimples showed no crack on the particle surface and fairly preserved its shape. The Al matrix around the particle was heavily deformed and another microvoid population was observed in the matrix. It is noteworthy that such characteristic of fracture mode is similarly observed regardless of deformation temperature from room temperature to 723 K. However, it was also importantly observed that the dimples of high deformation temperature (a) were deeper and larger than those formed at room temperature (b).

To clarify the crack propagation behavior of tensile deformation, cross-sectional observation was performed on tensile fracture surfaces at room temperature and 623 K, with the results shown in Fig. 9. Subsequently, the EPMA analysis results of the elements Al, Mg, Si, Co and Ni around the fracture surfaces are shown in Fig. 10. In Fig. 9, a gradient of roughly 45° of the fracture surface to the tensile direction was observed. The location and distribution of the \((\text{Ni}, \text{Co})_3\text{Al}_4\) particles and pores (containing segregated Mg and Si elements) around crack path could be well observed in Fig. 9 and Fig. 10. Regardless of the deformation temperature, those cracks could not shear the \((\text{Ni}, \text{Co})_3\text{Al}_4\) phase in the process of crack propagation. As a result, the deflection of crack path by the reinforcing particle was clearly detected. Conversely, the cracks developed easily along the pores. In addition, particle-matrix interfacial decohesion could not be found in all of the samples, which confirms the good interfacial bonding between the Al matrix and the \((\text{Ni}, \text{Co})_3\text{Al}_4\) particle. Thus, it seems to be reasonable to assume that the superiority of high temperature tensile properties of new Al-Mg-Si-CoNi alloy is mainly due to the fine particle size, good particle-matrix interfacial bonding, uniform distribution of the reinforcement particle, and the maintenance of strengthening role of \((\text{Ni}, \text{Co})_3\text{Al}_4\) particle up to high temperature.
4. Conclusions

(1) Using powder ball milling, induction melting and subsequent continuous casting, it was possible to manufacture the Al-1%Mg-1.1%Si-0.8%CoNi alloy with 1–8 μm (Ni, Co)₃Al₄ phases.

(2) The microstructure of Al-1% Mg-1.1% Si-0.8% CoNi alloy was analyzed through XRD, SEM, FIB and HR-TEM. The new alloy consisted of a primarily Al matrix, (Ni, Co)₃Al₄ phase particles (reinforcing phase) and pores. The reinforcing phases were homogeneously distributed at an average distance of 17.4 μm, with an incoherent interface with the matrix.

(3) The new heat-resistant alloy (compared to conventional Al alloy) represented to have superior tensile properties at high temperatures, with almost no decrease in tensile strength (>623 K). The tensile strength and elongation at room temperature were 255.4 MPa and 15.7%, respectively. At 723 K, a tensile strength of 205.4 MPa and elongation of 20% indicated excellent heat resistance.

(4) A typical ductile fracture mode with dimples was observed on the high temperature tensile fracture surface. Inside the dimples, the (Ni, Co)₃Al₄ particle remained. At both room and high temperatures, the role of reinforcing particles was well maintained. The (Ni, Co)₃Al₄ particle impeded further development of the cracks, contributing to the increased tensile strength at high temperatures. In contrast, interior pores acted as an easy path for crack propagation.

Fig. 9 SEM observation results of cross-sectional tensile specimen, tested at 298 K (a) and 623 K (b).

Fig. 10 Cross-sectional EPMA analysis result of high temperature (623 K) tensile fractured sample.
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REFERENCES