Development of High-Strength Bolt Material of Al–Mg–Si Alloy by ECAP and Various Aging Treatments^{*1}

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At present, the strength of most of Al–Mg–Si alloy bolts is not enough, and thus highly expected to be increased, for example, to >500 MPa in ultimate tensile strength (UTS). In this study, mechanical strength of A6056 Al–Mg–Si alloy was aimed at improving, without diminishing the elongation, by combined application of equal-channel angular pressing (ECAP) and various aging treatments. Multi-pass ECAP and pre-aging (PA) treatment at 373 K for 1.2 ks was found to be effective in strengthening the bolt material subject to room-temperature storage followed by artificial aging. Especially, PA plus ECAP 2pass treatment exerted the biggest impact on the strength (i.e. 514 MPa in UTS) with a reasonable elongation to fracture (i.e. 16%). Such a high strength and good ductility exceeds minimum requirement for aluminum-made bolts registered in JIS B1057, and thus the developed PA-ECAP 2pass specimen can be utilized potentially as a high-strength bolt material.

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1. Introduction

In recent years, CO₂ emission regulation has been imposed on automobiles to prevent global warming, and thus automobile manufactures eagerly deal with the problem by improving efficiency of internal-combustion engines, by developing new power sources and by replacing conventional materials with lighter weight aluminum or magnesium alloys. In such a trend, materials of bolts which fasten automobile components are also paid attention, and a demand for aluminum-made bolts is remarkably increasing. This is because galvanic corrosion caused by the difference between standard electrode potentials occurs if aluminum or magnesium components are fastened by conventional steel bolts. The application of aluminum-made bolts can solve this corrosion problem¹⁾ in addition to a further reduction of weight of automobiles. However, ultimate tensile strength (UTS) of conventional corrosion-resistant and formable Al–Mg–Si alloy bolts is about 320 MPa (Table 1²⁾), and thus the range of application is quite limited, resulting in plastic region tightening disallowing the reuse of bolts. Therefore, if further strengthening can be achieved, elastic region tightening becomes possible, enabling not only to reuse the bolts but also to simplify working procedure of fastening. Recently, it was reported that forging workability of a relatively high-strength A6056 alloy can be improved with 420 MPa in UTS by reexamining the conditions of casting and wire drawing.³⁾ However, strengthening is still in demand, therefore new methods such as severe plastic deformation have been attempted.

Equal-Channel Angular Pressing (ECAP)^{4–6)} is one of severe plastic deformation methods to obtain ultrafine grains at submicrometer or nanometer scale. A rod-shaped specimen

Table 1 Minimum requirement of mechanical properties for aluminum-made bolts. Six alloys registered in JIS B1057²⁾ are compared.

Symbol	Alloy	UTS (MPa)	0.2%YS (MPa)	Elongation (%)
AL1	A5052	250–270*	180–230*	3–4
AL2	A5056	280–310*	200–205*	6
AL3	A6061	310–320*	250–260*	7–10
AL4	A2024	380–420*	260–290*	6–10
AL5	A7N01	460	380	7
AL6	A7075	510	440	7

*Depends on bolt size

is pushed into a die with a bent channel, and is subjected to shear deformation without changing the cross-sectional shape. It is possible to introduce a large amount of strain by repeating this process; i.e. by increasing the number of passes. Hockauf *et al.*⁷⁾ developed an A6056 alloy material with ~450 MPa in UTS by combined application of ECAP 2pass and aging treatment at 473 K. Jin *et al.*^{8,9)} also succeeded in producing high-strength bolts of A6061-T6 alloy with >460 MPa in UTS by means of a newly developed and continuously repeatable spring-loaded ECAP. Therefore, it seems that the development of bolt materials with >500 MPa in UTS will be possible if both of ECAP and aging treatments are appropriately applied to Al–Mg–Si alloys.

In general, Al–Mg–Si alloys are utilized as structural components because of their good age-hardenability, but it is well known that if stored at room temperature (RT) before artificial aging, negative effect of two-step aging degrades the age-hardenability.¹⁰⁾ Pre-aging (PA) before RT storage can overcome such a negative effect of two-step aging,¹¹⁾ and thus is expected to similarly work for Al–Mg–Si alloy bolts because their manufacturing process includes RT storage.

Therefore, the purpose of this study is to increase the mechanical strength of an A6056 Al–Mg–Si alloy, without

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Table 2 Chemical composition of the investigated A6056 Al–Mg–Si alloy (mass%).

Mg	Si	Cu	Fe	Mn	Zn	Cr	Ti+Zr	Al
0.88	1.02	0.71	0.16	0.58	0.17	0.03	0.15	Bal.

diminishing the elongation, for developing a high-strength bolt material through grain refinement strengthening by ECAP and precipitation strengthening by various aging treatments including PA.

2. Experimental Procedure

Cu-added A6056 Al–Mg–Si alloy with a chemical composition in Table 2 was investigated in this study. Rod-shaped specimens with a diameter of 7.9 mm and a length of 45 mm were fabricated by swaging and cutting. The conventional manufacturing process of aluminum-made bolts consists of cold forging (screw head forming), solid solution treatment, water quenching (WQ), natural aging (RT storage), artificial aging and thread rolling. In this study, however, the specimens subject to processes excluding forging and thread rolling were referred to as the conventionally processed (CP) specimen. The schematic representation of the thermomechanical procedure of the CP specimen is shown in Fig. 1(a), in which commercially adopted conditions of natural and artificial aging are depicted as at RT for 86.4 ks and at 448 K for 28.8 ks, respectively. The ECAP, PA and PA-ECAP specimens were fabricated by 1, 2 or 4 passes of ECAP within 7.2 ks after WQ (Fig. 1(b)), by pre-aging at 373 K for 1.2 ks immediately after WQ (Fig. 1(c)) and by combined application of PA and ECAP (Fig. 1(d)), followed by artificial aging at 373, 408 or 448 K. It can be considered

that there is no influence of timing of ECAP on mechanical properties of the ECAP and PA-ECAP specimens because the hardness of the two specimens does not change until 7.2 ks of RT storage after WQ, and until 24 h of RT storage after ECAP.

The utilized die for ECAP possesses a L-shaped bending channel with a channel angle of $\Phi = 90^\circ$, an outer-corner angle of $\Psi = 30^\circ$ and a diameter of 8 mm. The amount of shear strain ε_N after ECAP N pass is given by eq. (1).¹²⁾

$$\varepsilon_N = N \left[\frac{2 \cot\left(\frac{\Phi}{2} + \frac{\Psi}{2}\right) + \Psi \operatorname{cosec}\left(\frac{\Phi}{2} + \frac{\Psi}{2}\right)}{\sqrt{3}} \right] \quad (1)$$

The extrusion rate was set at 0.067 mms^{-1} and the specimen was rotated by 180° for each pass (Route C⁴⁾) because introduced strain after ECAP 2pass is more homogeneous and symmetrical than without rotation or Route Bc, in which the specimen is rotated by 90° for each pass.

Vickers hardness test, tensile test, transmission electron microscopy (TEM) observation and differential scanning calorimetry (DSC) measurement were conducted for the specimens subject to various thermomechanical procedures. The age-hardenability of each specimen was measured by a Vickers hardness tester (Matsuzawa MMT-X1) with a load of 4.9 N for a dwelling time of 15 s. The hardness was determined as an average value of five tested points out of seven ones by excluding the maximum and minimum values. The cross-section of rod-shaped specimens perpendicular to their longitudinal direction was tested for the ECAP and PA-ECAP specimens.

Tensile test was performed using a universal testing machine (Instron MODEL 4487) with an initial strain rate of $2.8 \times 10^{-3} \text{ s}^{-1}$ at RT. The tensile test samples possess a dumbbell shape with a parallel length of 18 mm and a

Fig. 1 Schematic representation of the applied thermomechanical procedures to (a) conventionally processed (CP), (b) ECAP, (c) pre-aged (PA) and (d) PA-ECAP specimens.

Fig. 2 Attained hardness (a) and the corresponding TEM microstructures of the (c) ECAP 1pass, (d) ECAP 2pass and (e) ECAP 4pass specimens. The TEM specimens were prepared from disk-shaped thin films after ECAP as shown in (b).

diameter of 3 mm. After threaded at grip parts, two punch marks with an interval of 15 mm were made at parallel part, and then measured again after tensile test to quantify the change of the interval with a profile projector. In case that the interval cannot be measured with the profile projector, a caliper was utilized instead.

The foils for TEM observation were punched out from the center of the rod-shaped ECAP and PA-ECAP specimens (Fig. 2(b)), and then thinned by a twin-jet electropolishing unit (Struers Tenupol-5) with methyl nitrate (nitric acid:methanol = 85:15) cooled at 253 K. Bright- and dark-field images were observed using a JEM-2100F microscope at an accelerating voltage of 200 kV.

Disc-shaped samples for DSC measurement were cut from the rod-shaped specimens with a weight of 36 mg. ULVAC Riko DSC-RL was utilized to measure heat flux during heating at a heating rate of 0.17 K s^{-1} with a reference counterpart of high-purity aluminum of 99.99 mass%.

3. Results and Discussion

3.1 Improvement of strength by ECAP

Figure 2(a) shows the change in Vickers hardness by ECAP 1, 2 and 4passes (i.e. $\varepsilon_1 = 1.02$, $\varepsilon_2 = 2.03$, $\varepsilon_4 = 4.06$). The attained hardness increased from HV74 of the ECAP 0pass specimen to HV146 (i.e. $\Delta \text{HV}72$) by ECAP 1pass,

HV155 (i.e. $\Delta \text{HV}81$) by ECAP 2pass and HV165 (i.e. $\Delta \text{HV}91$) by ECAP 4pass, respectively. This is because a reduction in grain size has occurred as illustrated in the corresponding TEM microstructures (Fig. 2(c)–(e)). For example, the thickness of grains elongated to 45° from the extrusion direction⁴⁾ was decreased from $\sim 122 \text{ nm}$ (ECAP 1pass) to $\sim 90 \text{ nm}$ (ECAP 4pass), suggesting that grain refinement strengthening is activated with increasing the amount of strain introduced by ECAP (The thickness of grains was measured by the so-called line intercept method). The contribution of dislocation strengthening was assumed to be equivalent in each specimen because quantitative data on dislocation densities was not obtained in this study.

Figure 3(a) shows the changes in Vickers hardness during artificial aging at 448 K for the CP, ECAP 1, 2, and 4pass specimens. Although the increment in hardness was decreased from $\sim \Delta \text{HV}65$ (CP) to $\sim \Delta \text{HV}14$ (ECAP 1pass) or $\sim \Delta \text{HV}9$ (ECAP 2pass), the hardness of the ECAP 1 and 2pass specimens was increased by artificial aging, but the hardness of the ECAP 4pass specimen did not increase at all at 448 K (Fig. 3(b)). Such a suppressed age-hardening is due to coarsely and sparsely formed grain boundary precipitates of the stable β phase as illustrated in Fig. 4. Note that both of the promoted diffusion of solute atoms along grain boundaries and the decreased activation energy required for the heterogeneous nucleation¹³⁾ are attributed

Fig. 3 Changes in (a) Vickers hardness and (b) increment of Vickers hardness during artificial aging at 448 K for the CP and ECAP 1, 2 and 4pass specimens.

Fig. 4 TEM microstructures of the ECAP 2pass specimen after aged at 448 K for 1.8 ks. The stable β phase is heterogeneously precipitated at grain boundaries as shown by arrows.

to such a unfavorable formation of β , resulting in the suppressed formation of transgranular precipitates of the strengthening β'' phase. Therefore, it was found that concurrent strengthening by grain refinement and artificial aging is hardly achieved at a conventional aging temperature of 448 K.

Figure 5 shows the changes in Vickers hardness during artificial aging at 373, 408 and 448 K for the CP, ECAP 1, 2 and 4pass specimens. Although the age-hardening rate was decreased, both of the increment in hardness and the attained hardness increased with decreasing aging temperature. Therefore, based on the authors' proposal on concurrent strengthening by grain refinement and artificial aging,¹⁴⁾ the lowering of aging temperature was confirmed again to be effective even for the ECAP specimens of A6056 alloy utilized in this study.

3.2 Improvement of strength by pre-aging

Figure 6(a) shows the changes in Vickers hardness during artificial aging at 448 K for the CP, WQ and PA specimens. The WQ specimen was subjected to artificial aging immediately after water quenching without RT storage. The hardness of the CP specimen after artificial aging for 28.8 ks (HV134) was smaller than that of the WQ specimen (HV144), and the age-hardening rate up to 28.8 ks was also decreased in the CP specimen. This result suggests that the negative effect of two-step aging is exerted on the age-hardenability of the investigated A6056 alloy. On the other hand, the PA specimen subject to pre-aging at 373 K for 1.2 ks recovered the suppressed age-hardening rate even after RT storage for 86.4 ks, and achieved almost the same attained hardness (HV143) as that of the WQ specimen (HV144). Therefore, it was confirmed that the application of pre-aging can increase the attained hardness (i.e. improvement of HV9), or shorten the commercially adopted aging time (i.e. from 28.8 ks to $\sim$ 10 ks at which the equivalent hardness of HV134

Fig. 5 Change in Vickers hardness during artificial aging at 373 to 448 K for the CP and ECAP 1, 2 and 4pass specimens. The aging conditions applied to tensile test specimens in Fig. 11 are indicated by circles.

Fig. 6 Changes in Vickers hardness during artificial aging at (a) 448 K and (b) 373 K for the CP, WQ (Immediately aged after water-quenched without natural aging) and PA specimens.

to the conventional A6056 alloy bolts can be obtained) of the CP specimen. The similar recovery of age-hardening rate of the CP specimen was also observed at 373 K in Fig. 6(b), where the PA specimen shows similar age-hardening behavior as the WQ specimen. Therefore, pre-aging was found to be effective even at lower artificial aging temperatures.

3.3 Improvement of strength by pre-aging and ECAP

Figure 7(a), (b) shows the changes in hardness during artificial aging at 373 K for the ECAP 1, 2pass and PA-ECAP 1, 2pass specimens. By the application of pre-aging before ECAP, the hardness of the PA-ECAP specimen became higher than that of the ECAP specimen, but the increment in hardness was monotonously decreased from $\Delta HV10$ (ECAP 1pass) to $\Delta HV4$ (ECAP 2pass) or $\Delta HV2$ (ECAP 4pass, but no data is shown in the paper). This decrease of increment in hardness is probably because nanoclusters formed during pre-aging were decomposed and re-dissolved into the matrix by accumulative dislocations introduced by the subsequent ECAP, resulting in the gradual decrease in contribution of nanoclusters with increasing the number of ECAP passes. Note that the difference in hardness between WQ and PA specimens (i.e. $\Delta HV29$. See Fig. 6(a), (b)) is due to the formation of nanoclusters, and thus if the amount of nanoclusters is decreased by ECAP, the increase in hardness from ECAP specimens must have been decreased to $\Delta HV10$, $\Delta HV4$, $\Delta HV2$ and so on.

On the other hand, the age-hardenability of the PA-ECAP 1pass specimen ($\Delta HV14$) was lower than that of the ECAP 1pass specimen ($\Delta HV18$) as illustrated in Fig. 7(c). This suggests that the amount of precipitates formed during artificial aging is smaller than that of the ECAP 1pass specimen because a part of nanoclusters formed during pre-aging survive even after ECAP 1pass, resulting in the suppressed age-hardening during artificial aging of the PA-ECAP 1pass specimen. However, the age-hardenability of the PA-ECAP 2pass specimen ($\Delta HV14$) was higher than that of ECAP 2pass specimen ($\Delta HV12$) as illustrated in Fig. 7(d). This reverse phenomenon may be because most of nanoclusters formed during pre-aging are decomposed and re-dissolved into the matrix by ECAP 2pass, and thus a larger

Fig. 7 Changes in (a, b) Vickers hardness and (c, d) increment of Vickers hardness during artificial aging at 373 K for the ECAP 1 pass and PA-ECAP 1 pass specimens (a, c), or for the ECAP 2pass and PA-ECAP 2pass specimens (b, d). The aging conditions applied to tensile test specimens in Fig. 11 are indicated by circles.

Fig. 8 TEM microstructures of the PA-ECAP 2pass specimen after peak-aging at 373 K for 345.6 ks. Strain contrast arising from nanoclusters is visible within the matrix, together with the needle-shaped β'' phase at dislocations and grain boundaries.

Fig. 9 DSC curves of the AQ (Immediately heated after water-quenched without natural aging), PA and PA-ECAP 2pass specimens.

amount of precipitates can be formed during artificial aging, responsible for the larger increment in hardness of the PA-ECAP 2pass specimen. As the corresponding TEM microstructures (Fig. 8) cannot prove the existence of such nanoclusters and/or precipitates, the validity of the above mechanism was evaluated by DSC measurement as described below.

Figure 9 shows the DSC curves of the as-quenched (AQ), PA and PA-ECAP 2pass specimens. According to the results of previous studies,^{15–20)} the four exothermic peaks A, B, D and E can be regarded as the formations of nanoclusters, GP zones or Cu-containing clusters, the β'' phase, and the β' and Q' phases (i.e. precursor of the quaternary Q - $Al_5Cu_2Mg_8Si_6$ phase), respectively, whereas the endothermic peak C around 473 K as the dissolution of nanoclusters. It is clearly seen in Fig. 9 that the peak A indicating the formation of nanoclusters disappeared in the PA specimen, but appeared again in the PA-ECAP 2pass specimen. This suggests that nanoclusters have been formed by pre-aging, but decomposed and re-dissolved into the matrix by ECAP 2pass, as predicted by the above proposed mechanism. Further investigation using three-dimensional atom probe is in progress to clarify the effect of accumulative dislocations

Fig. 10 Attained hardness of the investigated specimens with various ECAP and aging treatments. The contribution of each thermodynamic procedure to hardness increase is also indicated.

Fig. 11 Stress-strain curves of the newly developed bolt material with various ECAP and aging treatments. The curve of the CP (aged at 448 K for 28.8 ks) specimen is also shown for comparison.

introduced by ECAP on the distribution of nanoclusters formed during pre-aging.

3.4 Mechanical properties of developed bolt material

Figure 10 shows the contribution of each thermodynamic procedure to the attained hardness of the developed bolt material in this study. The attained hardness of every ECAP specimen was quite higher than that of the CP specimen aged at 448 K for 28.8 ks (i.e. HV134 as a standard hardness of the conventional A6056 alloy bolt). Especially, the attained hardness of the PA-ECAP 2pass and ECAP 4pass specimens successfully exceeded HV170 owing to the greater contribution of ECAP.

Figure 11 shows the results of tensile test for the developed bolt material. The estimated UTS of the ECAP 1, 2 and 4pass specimens; i.e. 462, 492 and 509 MPa, was higher than that of the CP specimen (405 MPa), but the elongation to failure was monotonically decreased from 17% of the CP specimen to 12% of the ECAP 4pass specimen (Table 3). However, UTS and elongation to failure of the PA-ECAP 2pass specimen were 514 MPa and 16%, suggesting that combined application of pre-aging and ECAP 2pass can achieve not only a high strength comparable to that of the ECAP 4pass specimen, but also sufficient ductility through a “mild” process of ECAP 2pass.

Table 3 The obtained tensile properties after peak-aged at 373 K for the newly developed bolt material with various ECAP and aging treatments. The values of the CP (aged at 448 K for 28.8 ks) specimen are also listed for comparison.

	UTS (MPa)	0.2%YS (MPa)	Elongation (%)
CP (aged at 448 K for 28.8 ks)	405	352	17
ECAP 1pass	462	432	16
ECAP 2pass	492	465	14
ECAP 4pass	509	493	12
PA-ECAP 2pass	514	491	16

Figure 12 shows strength-ductility balance of the developed bolt material in this study. For comparison, six registered alloys for aluminum-made bolts (Table 1²⁾) and previously reported Al–Mg–Si alloys subject to ECAP^{7–9,21–23)} were also plotted. The PA-ECAP 2pass specimen was found to possess quite high strength comparable to that of A7075 alloy with much improved ductility. Therefore, combined application of pre-aging and ECAP has proved that the strength of A6056 alloy can be increased to >500 MPa (i.e. 514 MPa in UTS) with a reasonable elongation to fracture (i.e. 16%). Such a high strength and good ductility exceeds minimum requirement for aluminum-made bolts registered in JIS B1057, and thus the developed PA-ECAP 2pass specimen can be utilized potentially as a high-strength bolt material.

4. Conclusions

The age-hardening behavior of A6056 Al–Mg–Si alloy subject to pre-aging and equal-channel angular pressing (ECAP) was investigated, and the possibility to utilize as a high-strength bolt material was estimated based on the mechanical properties including Vickers hardness, ultimate tensile strength (UTS) and elongation to failure. The obtained results were summarized below.

- (1) Although the increment in hardness was drastically decreased, the hardness of the ECAP 1 and 2pass specimens increased during artificial aging at 448 K, but the ECAP 4pass specimen did not show any age-hardenability at 488 K. On the other hand, both of the increment in hardness and the attained hardness have been improved at 373 K, suggesting that concurrent strengthening by grain refinement and artificial aging can be achieved if artificial aging temperature is lowered.
- (2) The pre-aging at 373 K for 1.2 ks could overcome the negative effect of two-step aging appeared in the CP specimen. Therefore, the application of pre-aging was proved to be effective not only in increasing the attained hardness but also in shortening the commercially adopted aging time to ~10 ks at which the equivalent hardness to the conventional A6056 alloy bolts can be obtained.
- (3) The PA-ECAP 2pass specimen possessed a good strength-ductility balance of 514 MPa in UTS and

Fig. 12 Strength-ductility balance of the newly developed bolt material with various ECAP and aging treatments. The mechanical properties of previously reported ECAP-processed specimens^{7–9,21–23)} and registered alloys for aluminum-made bolts (JIS B1057²⁾) are also plotted for comparison.

16% in elongation to fracture. These values exceed minimum requirement for aluminum-made bolts registered in JIS B1057, and thus the developed specimen can be utilized potentially as a high-strength bolt material.

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