# Influence of Solute Atoms on Thermal Stability of Fibrous Structure in 2024 Aluminum Alloy Extrusion

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The influence of the cooling rate after homogenization heat treatment and the heating temperature of the ingot before extrusion on the thermal stability of the fibrous structure of a 2024 aluminum alloy extrusion after T4 temper was investigated. It became apparent that the thermal stability of the fibrous structure was influenced by both the main elements (copper and magnesium) and the transition element (manganese). The fibrous structure was stabilized when the solute atoms of copper, magnesium and manganese were increased by rapid cooling after the homogenization heat treatment and the extrusion temperature was close to the nose temperature of the manganese precipitation. Therefore, the thermal stability of the fibrous structure decreased when the copper and magnesium were not present or the manganese amount was reduced by half.

Keywords: aluminum alloy, 2024, extrusion, fibrous structure, recrystallization

## 1. Introduction

The 2024 aluminum alloy has been used as a structural material, e.g., in aircraft, for about 80 years. The homogenization heat treatment of this alloy is generally carried out between 450 and 500 °C because its solidus temperature is 502 °C [1, 2]. The 3003 aluminum alloy contains manganese in the range between 1.0 to 1.5 mass%, and the nose temperature of manganese precipitation is about 500 °C. Thus, the microstructure of the extruded products becomes fibrous due to the fine Al-Mn(Fe)-Si compounds that precipitated during the homogenization when the homogenization heat treatment is carried out at 500 °C [3]. In a similar way, the 2024 aluminum alloy also contains manganese in the range between 0.30 to 0.9 mass%, and the extruded products tend to form a fibrous structure because of the fine compounds, e.g., Al-Cu-Mn(Fe), precipitate at the above-mentioned homogenization temperature [4]. However, abnormal grain growth occurs and it decreases the material strength when the process conditions are not appropriate. Therefore, stabilizing the fibrous structure is an important issue. In this study, the influence of the cooling condition after homogenization heat treatment and the pre-heating temperature before extrusion on the microstructure of a 2024 aluminum alloy extrusion is described.

## 2. Experimental procedure

The 2024 aluminum alloy billets of 90-mm diameter and 100-mm length with the composition of Al-4.5mass%Cu-1.7mass%Mg-0.6mass%Mn were prepared by DC casting. The billets were homogenization heat treated at 490 °C for 10 h, and cooled by water quenching (WQ), air cooling (AC; 300 °C/h), and furnace cooling (FC; 30 °C/h). After homogenization, the billets were pre-heated at 350, 400 or 450 °C using an induction furnace, and then extruded. The size of the extrusions were 3.0 x 35 mm. The extruded samples will be expressed by the cooling condition after the homogenization and the pre-heating temperature before extrusion, that is, WQ-350 °C means that the billet was water quenched after homogenization and pre-heated at 350 °C, and FC-450 °C means the billet was cooled in the furnace after homogenization and pre-heated at 450 °C. The extruded samples were solution heat treated at 490 °C for 1 h by using an air-furnace, and obtained T4 temper. Observations of the optical microstructures and the electrical conductivity measurements by

Signatest equipment were carried out on the billets after the homogenization heat treatments. The electrical conductivity measurements, observations of the optical microstructures of L-ST cross sections at the width center, and observations of the transmission electron microstructure (TEM) of L-LT cross sections at the center of width and thickness were carried out on the as-extruded samples (F temper). Furthermore, the optical microstructures of the L-ST cross sections at the width center of the T4 tempered samples were observed. In addition, the influence of the manganese solutions on the thermal stability of the extruded samples was investigated as an additional experiment. In this experiment, the 2024 alloy billets were homogenization heat treated at 490 °C for 5 h, 10 h and 20 h, and then quenched in water. The billets were extruded at 450 °C, and then the extruded samples were solution heat treated in the same way as described. The optical microstructures of the T4 tempered samples were observed. Moreover, the influence of the main elements (copper and magnesium) and the transition element (manganese) on the thermal stability of the extrusions was investigated as a second additional experiment. In this experiment, the billets with the standard composition, Al-2.3mass%Cu-0.8mass%Mg-0.6mass%Mn, Al-0mass%Cu-0mass%Mg-0.6mass%Mn and Al-4.5mass%Cu-1.7mass%Mg-0.3mass%Mn, were prepared by the DC casting method, then homogenization heat treated at 490 °C for 10 h and quenched in water. The billets were extruded at 450 °C, and extruded into a 4.0 x 50mm shape. The extruded samples were solution heat treated at 490 °C for 1 h, and then the optical microstructures were observed.

## 3. Results and discussion

Figure 1 shows the optical microstructures of the ingots after the homogenization heat treatments. The constituent particles on the cell boundaries were divided and coagulated by the homogenization heat treatments in all samples, but the number density of the precipitates in the grains of the air-cooled sample in Fig. 1 (b) is greater than that of the water-quenched sample in Fig. 1(a). Moreover, because the size of the particles on the cell boundaries of the furnace-cooled sample in Fig. 1(c) is bigger than that of the air-cooled sample in Fig. 1(c) is bigger than that of the air-cooled sample in Fig. 1(b), it is apparent that the precipitation occurred not only in the grains, but also around the constituent particles on the cell boundaries. In addition, the measurement of the electrical conductivities suggested that the amount of the solute atoms decreased as the cooling rate decreased.



Fig. 1 Optical microstructures of the ingots after homogenization heat treatment. The cooling conditions after homogenization were: (a) water quench, (b) air cooling, and (c) furnace cooling. The electrical conductivities were: (a) 30.0 %IACS, (b) 40.3 %IACS, and (c) 43.6 %IACS.

Figure 2 shows the optical microstructures of the T4 tempered extrusion L-ST cross sections at the center thickness. A fibrous structure was observed in the WQ-450°C, WQ-400°C and AC-450°C samples, while an abnormal grain growth structure was observed in the other samples. That is, the fibrous structure was stabilized by the combination of the higher cooling rate after the homogenization heat treatment and the higher heating temperature before the extrusion. The samples with the fibrous structure also had abnormal grain growth structures in the surface regions to a depth of several hundred micrometers. The recrystallization could not be suppressed because the degree of shear stress is greater in the surface region. On the other hand, all the samples with F temper (as-extruded) had a fibrous structure. This fact suggests that the abnormal grain growth occurred during the solution treatment, and the difference in the microstructure of the T4 temper occurred because of the difference in the thermal stability of the fibrous structure.



Fig. 2 Optical microstructures of the samples in T4 temper.

The TEM structures of the as-extruded samples are shown in Fig. 3, and the changes in the electrical conductivity before and after the extrusion are shown in Fig. 4. Subgrain structures were observed in Fig. 3. The electrical conductivities of the FC-350°C and FC-450°C samples decreased due to the extrusions, while those of the WQ-450°C and WQ-350°C samples increased. Furthermore, the precipitates in the WQ-450°C sample were the largest, while those in the WQ-350°C sample were smaller and more abundant than in the WQ-450°C sample. This fact suggests that the precipitation condition during extrusion was different although the electrical conductivities were almost the same. The nose temperature of the precipitation of copper and magnesium (S' and S phases) is about 350°C,



Fig. 3 Microstructures of the extruded samples with F temper.



while that for manganese is about 450-500°C in the 2024 alloy. Thus the S' or S phases precipitate, while manganese is difficult to precipitate at 350°C. On the other hand, the S' and S phases partially dissolve and coagulate, and fine manganese-containing compounds precipitate at 450°C. It was reported that the fibrous structure of a Al-1.0mass%Si-0.6mass%Mg-0.6mass%Mn alloy became stabilized because of the precipitation of the Al-Mn compounds during a compression process over 350°C [5]. Based on the above mechanism, the changes in the microstructures of the samples in this study were postulated as described below, and are schematically shown in Fig. 5. Because of the changes in the electrical conductivity before and after extrusion, it was apparent that the copper and magnesium atoms precipitated during the extrusion of the WQ-450°C sample. Thus, the precipitates on the subgrain boundaries inhibited the recrystallization. In addition, manganese atoms precipitated during the extrusion because the temperature was close to the nose temperature of the manganese precipitation. The manganese precipitates, which formed during the homogenization heat treatment, tend to inhibit recrystallization. Besides that, manganese atoms precipitated on the subgrain boundaries also inhibited the recrystallization. The extruded sample had the fibrous structure because of the effects. Also, during the solution heat treatment process, S phases precipitated on the subgrain boundaries, while the finer Cu-Mg dissolved during heating. However, the fibrous structure was maintained after the solution heat treatment because of the manganese precipitates on the subgrain boundaries. On the other hand, the thermal stability of the fibrous structure of the WQ-350°C sample decreased compared to the WQ-450°C sample. Copper and magnesium atoms precipitated on the subgrain boundaries during extrusion, and they inhibited a recrystallization similar to the WQ-450°C sample. However, the degree of manganese precipitation was lower because of the lower temperature. Thus, abnormal grain growth occurred during the solution heat treatment because the manganese precipitation was not sufficient to inhibit recrystallization after the copper and magnesium on the subgrain boundaries dissolved. For the FC-450°C and FC-350°C samples, the amount of solute copper, magnesium and manganese atoms decreased because of the lower cooling rate after the homogenization heat treatments. Copper and magnesium partially dissolved just before extrusion, and precipitated on the subgrain boundaries during extrusion of the FC-450°C sample. However, the amount was smaller than for the WQ-450°C sample. In addition, the amount of the precipitation of manganese atoms on the subgrain boundaries became smaller than that of the WQ-450°C sample because of the smaller manganese solute atoms. Therefore, the thermal stability of the fibrous structure decreased similar to the WQ-350°C sample. For the FC-350°C sample, the precipitation of copper, magnesium and manganese was difficult to occur, therefore, the thermal stability of the fibrous structure decreased.



Fig. 5 Schematic model of the influence of the grain boundary precipitates on the thermal stability of the fibrous structure of the extrusions. The samples are (a) WQ-450°C, (b) WQ-350°C, (c) FC-450°C, (d) FC-350°C.

As mentioned above, it was considered that the stability of the fibrous structure during the solution heat treatment was influenced by the solute atoms of the main elements (copper and magnesium) and the transition element (manganese). The following results are verification of the assumption. Figure 6 shows the optical microstructures of the T4 tempered extrusions of which the homogenization heat treatment times of the used ingots were changed to 5h, 10h and 20h to clarify the effect of the solute manganese atoms. In Fig. 6, the samples of (a)-5h and (b)-10h showed the fibrous structure, while the (c)-20h sample showed an abnormal grain growth structure. This result suggests that the thermal stability of the fibrous structure decreased because the amount of solute manganese atoms decreased during the homogenization treatment and the amount of manganese precipitation during extrusion decreased when the homogenization time was longer. Figure 7 shows the optical microstructures of the T4 tempered extrusions of which the chemical compositions changed. The fibrous structure was obtained when the amount of copper and magnesium were half as shown in Fig. 7(b). However, the abnormal grain growth structure was obtained when the amount of manganese use atoms also obtained when the amount of copper and magnesium were half as shown in Fig. 7(c). In addition, the abnormal grain growth structure was also obtained when the amount of manganese was half as shown in Fig. 7(d). When copper and magnesium were free, the

type of the manganese-contain precipitates changed and the degree of inhibiting recrystallization may have changed. However, it is obvious that the thermal stability of the fibrous structure was influenced by both the main elements (copper and magnesium) and transition element (manganese), and their solute atoms had significant effects. In this study, it became clear that the thermal stability of the fibrous structure was influenced by the solute atoms of both the main element and the transition element.



Fig. 6 Effects of the homogenization heat treatment time of the billets on the stability of the fibrous structure after solution heat treatment: (a) 5h, (b) 10h, (c) 20h. The billets were quenched into water after the solution heat treatments.



Fig. 7 Effects of the chemical compositions on the stability of the fibrous structure after solution heat treatment: (a) Al-4.6mass%Cu-1.6mass%Mg-0.6mass%Mn, (b) Al-2.3mass%Cu-0.8mass%Mg-0.6mass%Mn,

(c) AI-0mass%Cu-0mass%Mg-0.6mass%Mn,

(d) AI-4.5mass%Cu-1.7mass%Mg-0.3mass%Mn.

#### 4. Conclusions

(1) It became clear that the thermal stability of the fibrous structure was influenced by both the main element (copper and magnesium) and the transition element (manganese). The fibrous structure was stabilized when the solute atoms of copper, magnesium and manganese were increased by the rapid cooling after the homogenization heat treatment and the extrusion temperature was close to the nose temperature of the manganese precipitation.

(2) The thermal stability of the fibrous structure decreased when the copper and magnesium were not present or the manganese amount was reduced by half.

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