THE EFFECT OF HEAT TREATMENTS ON PRECIPITATION BEHAVIOR OF DISPERSOIDS IN Al-Mg-Si-Mn ALLOY

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ABSTRACT

Precipitation-strengthening 6xxx alloys are widely used for building and construction and automobile industries due to their high strength, good weldability and corrosion resistance. However, their elevated-temperature properties (250–300°C) decrease rapidly due to the fast-coarsening of precipitates. On the other hand, dispersoid-hardening is one of the most efficient approaches to improve the elevated-temperature properties. Therefore, the evolution of dispersoids during heat treatment in Al-Mg-Si-Mn alloys was investigated in the present work. Various heat treatments with temperatures ranging from 300–500°C for up to 24 hours were performed. Electrical conductivity and microhardness were measured while optical and scanning electron microscopy combined with image analysis, were used to characterize the dispersoids. Special emphasis was placed on the evolution of the size and number density of the dispersoids under different heat treatment parameters. It was found that dispersoids started to precipitate at 350°C for 1 hour. Nucleation and growth were the main mechanisms at temperatures up to 400°C while coarsening occurred at temperatures above 450°C. The quantitative results indicated that dispersoids precipitation reached the optimum condition after treatment at 400°C for 2–4 hours with a fine size and high number density, resulting in the highest hardness.

KEYWORDS

6xxx aluminum alloys, Heat treatment, Dispersoid precipitation, Nucleation and growth, Coarsening

INTRODUCTION

The precipitation-hardening 6xxx aluminum alloys with major alloying elements of Mg and Si are one of the most widely used aluminum alloys for building and construction and automobile industries (Dinaharan & Murugan, 2012). In recent years, there have been increasing demands for aluminum structural components to be used at elevated temperatures (250–300°C) for different applications. However, the mechanical properties of 6xxx alloys at elevated temperature are typically reduced due to the rapid coarsening of precipitates at 250–300°C. It has been reported that the yield strength (YS) of AA6061 in the peak-aged condition drops from 276 MPa at room temperature to only 30 MPa at 315°C (Kaufman, 1999). Therefore, it is a key concern to improve the elevated-temperature properties of 6xxx alloys to expand their range of applications.

The dispersoid strengthening mechanism is reported to be one of the most efficient approaches to enhance the elevated-temperature properties in 3xxx alloys (Liu & Chen, 2015). Due to the formation of thermally stable dispersoids by applying appropriate heat treatments in AA3004 alloys, the YS at 300°C can be improved from 55 MPa to 78 MPa (Liu & Chen, 2015). Meanwhile, studies on the dispersoids in 6xxx alloys have also been performed. Lodgaard and Ryum (2000) investigated the precipitation behavior of dispersoids in 6xxx aluminum alloys during homogenization using a typical high temperature of 580°C. It was found that Mg played a significant role by inducing β'-Mg_2Si precipitates, which promoted the nucleation of dispersoids (Marioara, Andersen, Jansen & Zandbergen, 2001). The distribution of dispersoids was strongly dependent on the heating rate and temperature during heat treatment (Hu, Ogura, Tezuka, Sato & Liu, 2010). The heat treatment temperatures in the above cited papers were all at a high temperature (>530°C) with fixed holding time, where it is difficult to oversee the entire picture of dispersoid precipitation. Few systematic studies on the formation and evolution of dispersoids during heat treatment at a lower temperature range, such as 300–500°C, have been conducted, which is reported to be the precipitation temperature of dispersoids in 3xxx alloys (Liu & Chen, 2015).

In the present study, the precipitation behavior of dispersoids in Al-Mg-Si-Mn aluminum alloy subjected to various heat treatments has been studied through the evolution of electrical conductivity, microhardness and microstructure with special emphasis placed on the evolution of the size and number density of dispersoids. A preliminary assessment of the relationship between dispersoids distribution and mechanical properties was established in the present work.

EXPERIMENTAL METHODOLOGY

Two experimental Al-Mg-Si-Mn alloys (6082 type) were designed in the present work: Alloy A was the base alloy without Mn and the Alloy B had an addition of 0.52% Mn. Approximately 3 kilograms of material for each composition were batched in an electrical resistance furnace and cast in the permanent mold preheated at 250°C. The dimension of the cast ingots was 30 mm × 40 mm × 80 mm). The chemical compositions of the two experimental alloys are shown in Table 1.

Table 1. Chemical composition (wt. %) of the experimental alloys

Alloy	Fe	Mg	Si	Mn
A	0.26	0.99	0.66	**0.00**
B	0.27	1.07	0.67	**0.52**

As-cast samples were heat-treated at temperatures of 300, 350, 400, 450 and 500°C, using holding times from 1 to 24 hours, followed by water quenching. A constant heating rate of 100°C/h was applied. After heat treatment, samples were cut, mounted and polished for metallographic observation and image analysis. Electrical conductivity (EC) tests were conducted to estimate the level of precipitation of dispersoids. Before the EC tests, all the specimens were placed in the same area at room temperature for

over 30 minutes to stabilize the condition. Vickers hardness tests were performed to investigate the strengthening effect induced by dispersoids. A load of 10 g was applied with a dwelling time of 20 seconds. The indentations of hardness tests were made in the center of dendrites without crossing the dendrite boundaries and intermetallics. To characterize the evolution of dispersoids distribution for different heat treatment parameters, polished samples were etched with a 0.5% HF solution for 25 seconds to clearly reveal the dispersoid position. Images were obtained using an optical microscope (Nikon Eclipse ME600) and a scanning electron microscope (SEM, JEOL JSM-6480LV) equipped with energy dispersive spectrometer (EDS). Both number density and average diameter were performed based on SEM images to illustrate the evolution of dispersoids. The average diameter was obtained by measuring the main length of the dispersoids.

RESULTS AND DISCUSSION

As-Cast Microstructure

As-cast microstructures of the two alloys are shown in Figure 1. Microstructures of both alloys were similar, consisting of aluminum dendrite cells, dark primary Mg_2Si and gray Fe-rich intermetallics distributed at the dendrite boundaries. Based on the results of the image analysis, the volume fractions of primary Mg_2Si and Fe-rich intermetallics in Alloys A and B were similar, indicating that most of Mn in Alloy B remained in the aluminum matrix during solidification. This is further confirmed by the higher microhardness and lower EC in Alloy B in the as-cast condition, which was 67.0 HV and 34.9 %IACS compared with 62.5 HV and 47.7 %IACS for Alloy A. Therefore, it is reasonable to assume that most of Mn in Alloy B was in supersaturated solid solution of the aluminum matrix.
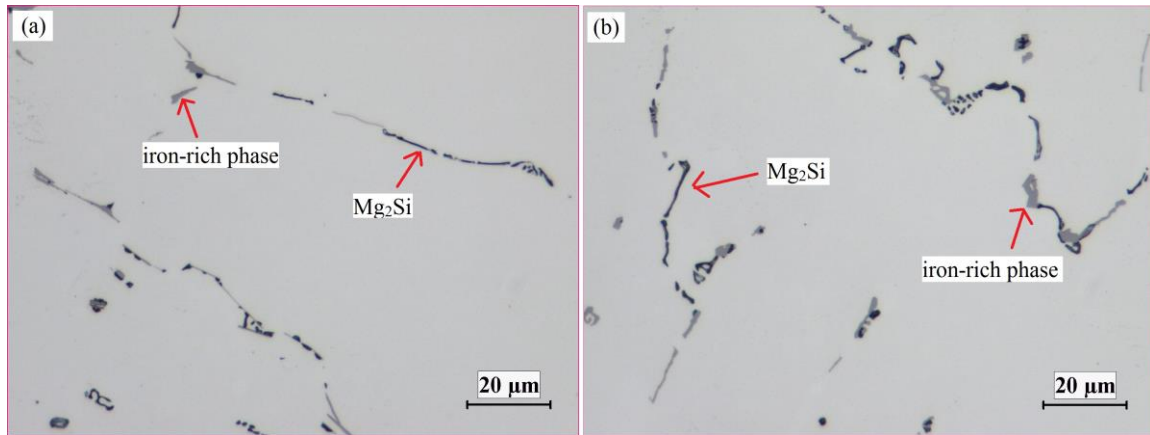

Figure 1. As-cast microstructures of Alloy A (a) and Alloy B (b).

Evolution of Dispersoids under Various Heat Treatments

Generally, EC is widely used to evaluate the changes of concentration of solute elements during heat treatment. The EC of experimental alloys can be described in the following equation (Liu & Chen, 2015):

$$1/EC_{Alloy-A}=0.0267+ 0.032\ Fe_{ss}\%+0.0068Si_{ss}\% + 0.003Mg_{ss}\% \qquad (1)$$

$$1/EC_{Alloy-B}=0.0267+ 0.032\ Fe_{ss}\%+ \mathbf{0.033Mn_{ss}\%}+ 0.0068Si_{ss}\% + 0.003Mg_{ss}\% \qquad (2)$$

where, $Fe_{ss}\%$, $Mn_{ss}\%$, $Si_{ss}\%$ and $Mg_{ss}\%$ are the weight percentages of the solute elements Fe, Mn, Si and

Mg. In the present work, the influence of other alloying elements on EC was negligible because the Fe, Si and Mg contents in two alloys were almost the same. Thus the change in the Mn solid solution level for Alloy B during heat treatment can be estimated by the EC difference between Alloy B and Alloy A (Eq. 3):

$$1/EC_{Alloy-B} - 1/EC_{Alloy-A} = 0.033Mn_{ss}\% \tag{3}$$

Similarly, the difference in Vickers hardness values between Alloy B and Alloy A can be used to estimate the strengthening effect of Mn-containing dispersoids in Alloy B.

Differences in 1/EC between Alloy A and Alloy B vary with temperature and holding time as shown in Figure 2a. When heat-treated at 300°C, differences in 1/EC between the two alloys was high and relatively stable with longer soak times, due to most of the solute Mn atoms remaining in the aluminum matrix in Alloy B. This is confirmed by the microstructures of the two alloys shown in Figure 3, where only large β-Mg$_2$Si precipitates were formed at 300°C in both alloys and no Mn-containing dispersoids were present in Alloy B.
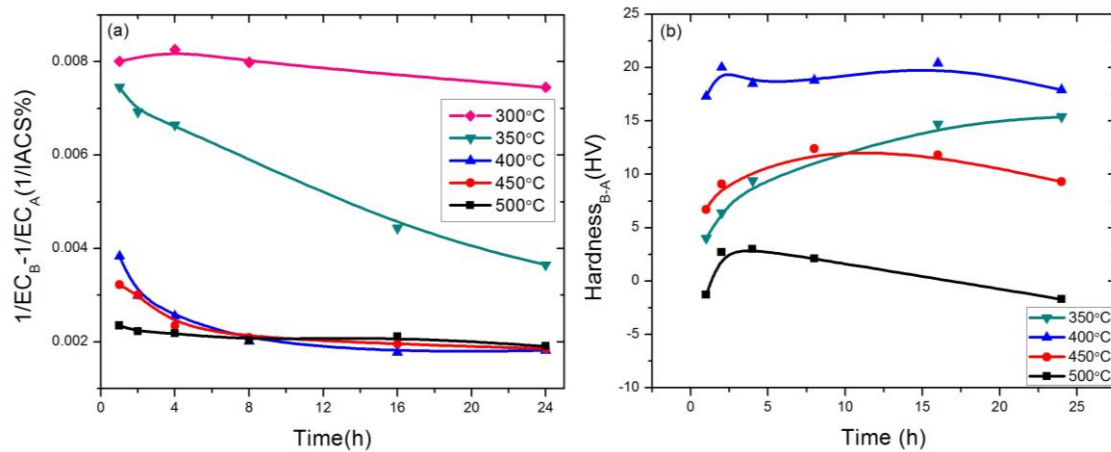

Figure 2. Differences in 1/EC (a) and Vickers hardness (b) between Alloy B and Alloy A as a function of heat treatment time and temperature
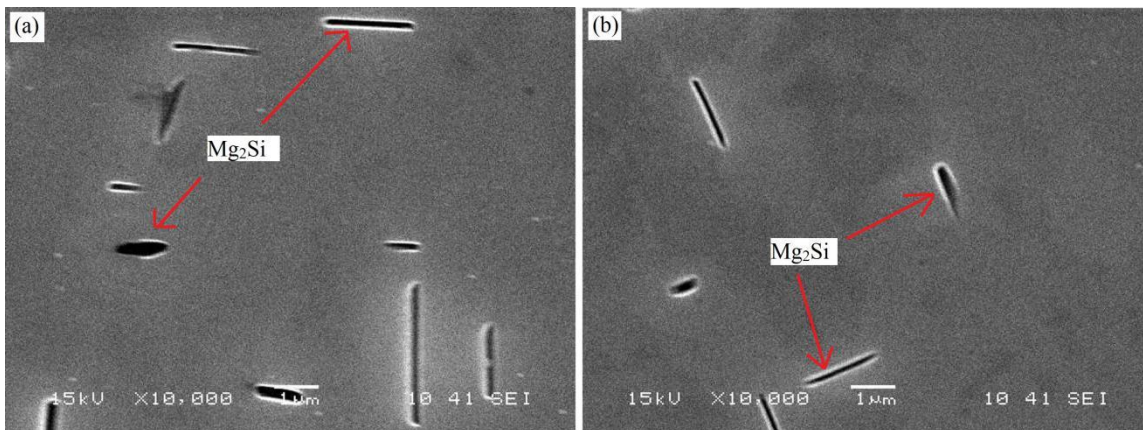

Figure 3. SEM images of Alloy A (a) and Alloy B (b) after heat treatment at 300°C for 24 hours

When the temperature was increased to 350°C, the difference in 1/EC between two alloys decreased significantly with longer holding time, indicative of a decreasing Mn solute concentration in the matrix. Figure 4 shows the microstructures of both alloys after a 350°C/24 h treatment. The black particles were observed in the aluminum matrix for alloy A (Figure 4a and 4c), which were identified as β-Mg_2Si by SEM based on the morphology and EDS analysis. Additionally, a large volume of fine dispersoids were observed in Alloy B (Figure 4b). Some dispersoid free zones were also observed around intermetallic particles at the dendrite boundaries. The evolution of the number density and size of dispersoids in Alloy B is shown in Figure 5. From Figure 5a it can be seen that a large number of fine dispersoids precipitated after 8 hours at 350°C and the number density of dispersoids increased with holding time, leading to an increase in microhardness with longer holding time (Figure 2b).
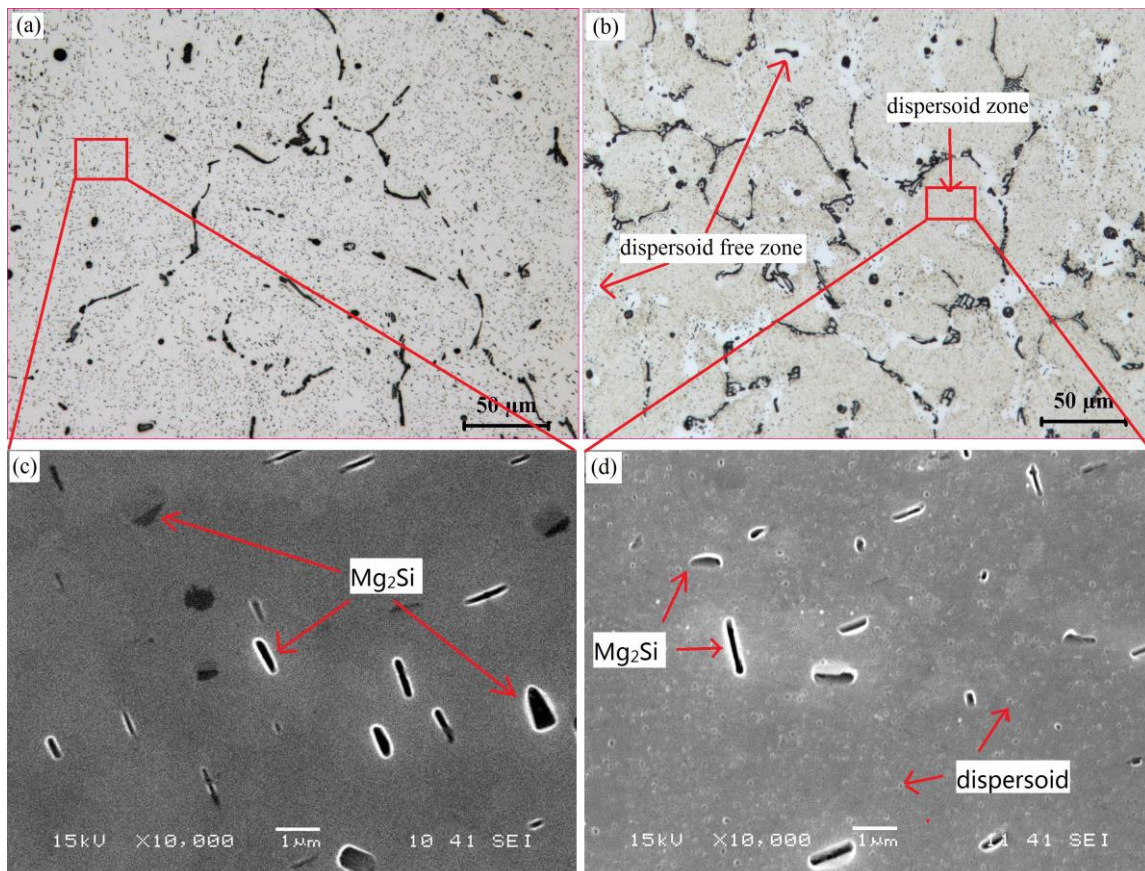

Figure 4. Microstructures of Alloy A (a, c) and Alloy B (b, d) after heat treatment at 350°C for 24 hours
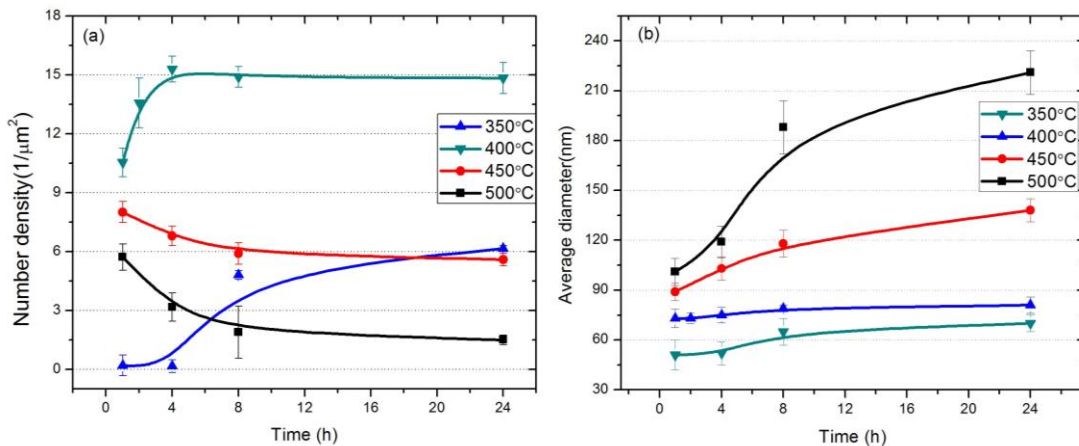
Figure 5. Evolution of the number density (a) and average diameter (b) of dispersoids in Alloy B as a function of time and temperature

As shown in Figure 2a, the differences in 1/EC at 400°C and 450°C were similar, and exhibited an initial fast drop followed by a plateau. However, the difference in microhardness at 400°C was higher than that at 450°C (Figure 2b), indicating a different evolution of the dispersoid structure at the two temperatures (Figure 6). No further images of Alloy A are shown in this paper due to the absence of dispersoids. As shown in Figures 6a and 6b, fine dispersoids were uniformly distributed in the matrix of Alloy B after 4 hours at 400°C with little change after exposure for 24 hours. This is in line with the quantitative results in Figure 5b, showing that at 400°C the dispersoid size remained fine up to 24 hours with a diameter of ~ 75 nm. However, the number density initially increased over the first 4 hours and then remained steady, indicating the principal operating mechanism of precipitation behavior of dispersoids at 400°C was still nucleation and growth. The high number density and fine size of the dispersoids made a significant contribution to the alloy properties (Hichem & Rebai, 2015), resulting in a high and stable hardness difference at 400°C (Figure 2b). It is apparent that dispersoids precipitation reached the optimum condition with relatively fine size and high number density after 2–4 hours at 400°C, leading to the highest difference in the hardness.
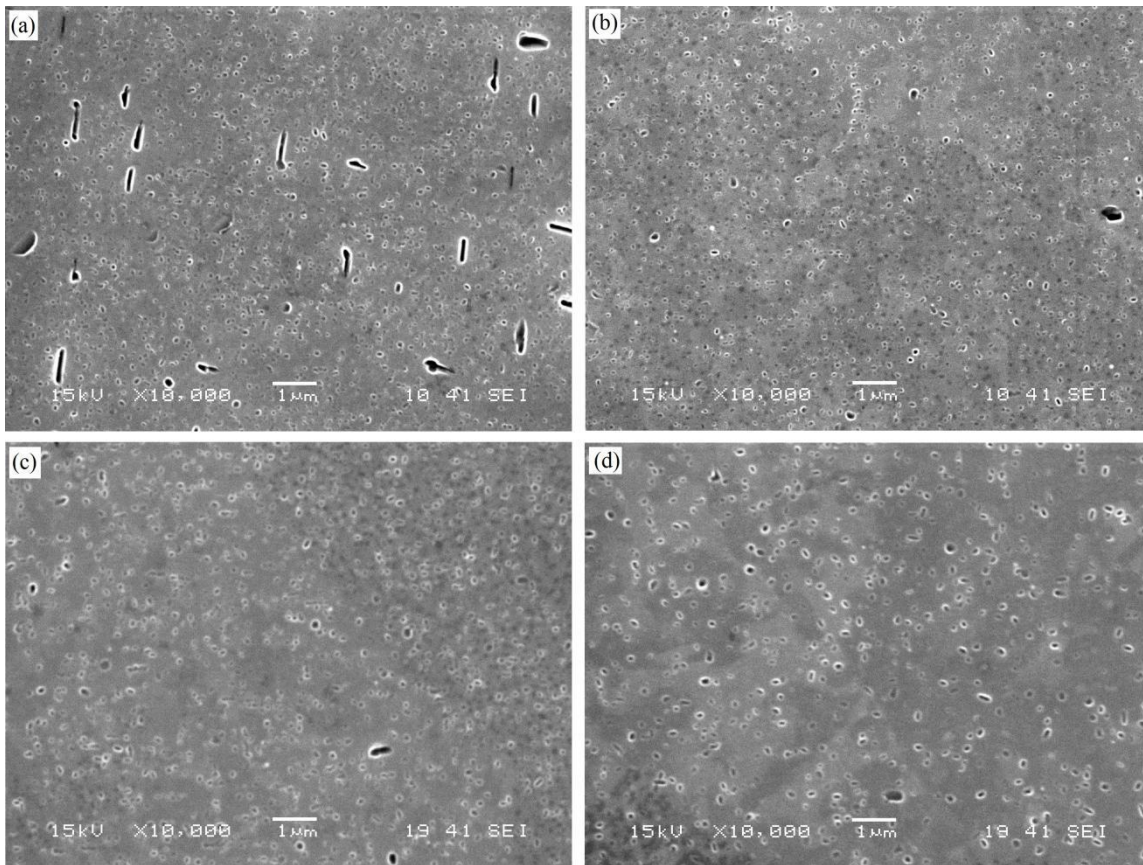
Figure 6. SEM images of Alloy B after heat treatment at 400°C for 4 hours (a) and 24 hours (b) and after heat treatment at 450°C for 1 hour (c) and 24 hours (d)

At 450°C (Figures 6c–d), a uniform dispersoid precipitate distribution was observed after 1 hour but the size increased and the number density decreased after 24 hours. Figure 5b shows that the dispersoid size increased quickly at this temperature, while the number density decreased with longer holding times, indicating that dispersoid coarsening occurred during holding at 450°C. Consequently, the hardness at 450°C increased over the first 8 hours due to the precipitation of dispersoids followed by a decrease, resulting from coarsening. Comparing dispersoids structures in Figure 6 and quantitative data in Figure 3, dispersoids were always finer and their number density higher at 400°C than that at 450°C, explaining the higher hardness difference at 400°C than at 450°C (Figure 2b).

When the temperature was increased to 500°C, the difference in 1/EC dropped for first 1−2 hours followed by a plateau (Figure 2a), indicating the fast decomposition of the Mn supersaturated solid solution to form dispersoids due to the higher diffusion rate at 500°C. Figure 7 shows the distribution of dispersoids at 500°C after 1 hour and 24 hour exposures respectively. Comparing Figures 7a and b with Figures 6c and d, it is apparent that dispersoids size at 500°C after 1 hour is larger than that at 450°C, while they are even much larger after 24 hours exposure, indicating fast coarsening at 500°C. This is also confirmed by Figure 5b where the size of dispersoids at 500°C was very large, in the range of 100–220 nm, while the number density was much lower, 2 μm^{-1} compared with a 15 μm^{-1} at 400°C. Correspondingly, the difference in hardness at 500°C in Figure 2b dropped rapidly with increasing holding time and it was the lowest of any condition due to the largest dispersoids with the lowest number density.
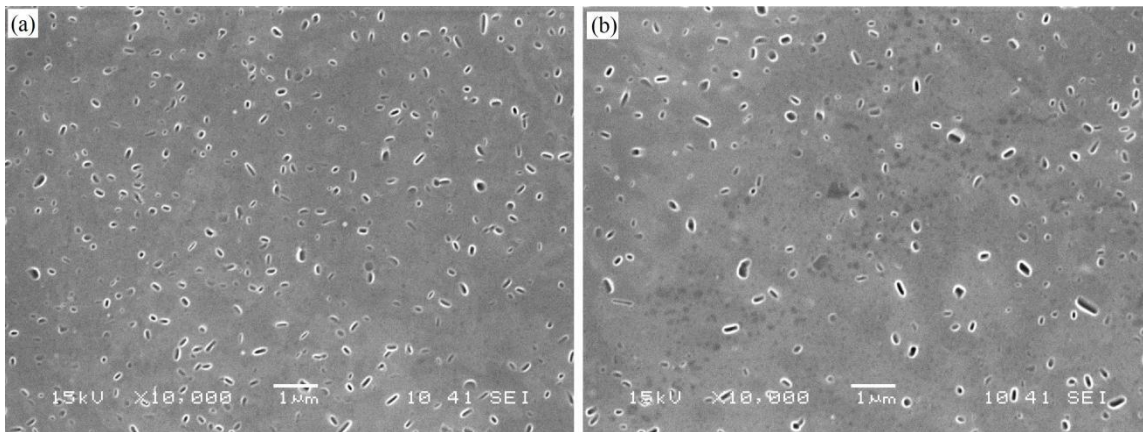
Figure 7. SEM images of Alloy B after the heat treatment at 500°C for 1 hour (a) and 24 hours (b)

In summary, as shown in Figures 2 and 5, dispersoids precipitation begins at 350°C and reaches the optimum condition after 2–4 hours at 400°C, which exhibits the maximum strengthening effect. However, when the temperature is increased to 450°C and 500°C, dispersoids coarsen with increasing holding time, leading to lower hardness. More systematic work, such as the detailed evolution of dispersoids (size and volume fraction) during heat treatment using a transmission electron microscopy as well as their influence on elevated-temperature properties will be performed in further work. In addition, the influence of alloying elements, such as Mn, Sc and Zr, on both microstructure and elevated-temperature properties will be investigated.

CONCLUSIONS

The precipitation behavior of dispersoids during heat treatment of an Al-Mg-Si-Mn (6082) aluminum alloy has been studied in the present work. It was found that dispersoids precipitation started at 350°C resulting in a fine particle size. At the slightly higher temperature of 400°C, the dispersoids size remained fine and the number density increased with increasing holding time. The evolution of the dispersoids distribution at 350–400°C was mainly dominated by nucleation and growth. When the temperature was further increased to 450 and 500°C, precipitation of dispersoids occurred more rapidly but their size became larger and the coarsening became the dominant mechanism. The optimum condition for dispersoids precipitation can be reached after 2–4 hours at 400°C, leading to the highest hardness of the material.

ACKNOWLEDGMENTS

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