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# Suppressed negative effects of natural aging by pre-aging in SiCp/ 6092Al composites

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# ABSTRACT

For most of Al-Mg-Si-(Cu) alloys and their composites, artificial aging hardening can be impaired by precedent natural aging. In this study, a pre-aging treatment before natural aging was adopted to improve the hardness of the artificially aged SiCp/6092Al composite and 6092Al alloy. Effects of the multi-step aging treatment, including pre-aging, natural aging and final artificial aging, on microstructures and mechanical properties were investigated by the hardness test, conductivity test, differential scanning calorimetry and transmission electron microscopy. It was shown that pre-aging exhibited a more obvious improvement in the final hardness in the composite, compared to that in the 6092Al alloy. The supersaturated solute atoms formed clusters during natural aging after pre-aging, but the clustering behaviors were different between the composite and 6092Al alloy, leading to the different pre-aging hardening effects. In the composite, it was easy for supersaturated solute atoms to participate into pre-aging clusters during natural aging. As pre-aging clusters could transform into precipitates during artificial aging, precipitate sizes were not affected by natural aging. In contrast, in the 6092Al alloy, it was easy for supersaturated solute atoms to form new clusters during natural aging after pre-aging. As it was difficult for these new clusters to transform into precipitates during artificial aging, precipitates were enlarged with prolonged natural aging time. Because the clustering behaviors during natural aging after pre-aging showed different effects on precipitation, the hardness after artificial aging did not change with prolonged natural aging time in the composite but decreased in the 6092Al alloy.

# 1. Introduction

SiC particles (SiCp) reinforced aluminum matrix composites have received significant attention in aerospace and defense applications due to their high modulus and strength, excellent wear resistance and fatigue properties [1-5]. Heat-treatable aluminum alloys (2xxx, 6xxx and 7xxx series alloys) are ideal matrices for aluminum matrix composites to achieve great load-bearing capabilities [3,6-9]. In general, these composites are applied after T6 treatment, which consists of solution, quenching and artificial aging (AA). However, the artificially aged composites often show poor ductility, which limits the cold plastic forming performance, such as quenching distortion correction and stamping.

The ductility of the composites is significantly affected by the matrix. Al-Mg-Si-(Cu) (6xxx series) alloys have high ductility and weak hardening in the natural aging (NA) state [10,11], which makes it possible for SiCp/6xxxAl composites to be plastically formed during NA, and

served after AA

The plastic forming requirements imply that SiCp/6xxxAl composites should be supplied sometimes in the NA state, so these composites will experience NA before AA. It was reported that for Al-Mg-Si-(Cu) alloys and their composites with Mg + Si > 1 wt%, AA hardness could be impaired by precedent NA. This phenomenon was called the negative NA effect [3,12-14].

After solution and quenching, solute atoms are supersaturated in the Al matrix. Due to the size mismatch between solute atoms and Al atoms, each solute atom will strain the Al matrix. In order to reduce energy, supersaturated solute atoms tend to form clusters by diffusion during NA. Due to the low NA temperature, short-range diffusion is dominant [15]. Clusters can be formed only based on neighboring atoms; hence, the composition distribution of NA clusters is discrete. It is commonly believed that the Mg/Si ratios of most NA clusters are far from 1. It is difficult for these clusters to act as nucleation sites or precursors of precipitates during AA, but they act as traps of vacancies, leading to the

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reduced vacancy concentration. As vacancies are important nucleation sites of AA precipitates [12,13,16,17], the nucleation of precipitates during AA is restricted. Partial precipitates preferentially grow at limited nucleation sites, resulting in enlarged average precipitate sizes and broadened precipitate size distribution, which impairs the hard-ening ability of AA [18,19].

Pre-aging (PA) treatments at temperatures above 70 °C are widely conducted immediately after quenching to improve the AA hardness of naturally aged Al–Mg–Si-(Cu) alloys [20–22]. The clusters formed during PA inhibit the clustering behavior during NA [17]. The high PA temperatures make long-range diffusion feasible. To achieve higher electronic and elastic stability, Mg/Si ratios of PA clusters are uniform and close to 1 [15]. It is widely accepted that these PA clusters can constitute precipitates during AA [16,17]; hence, PA can inhibit the negative NA effect and thus improve AA hardness.

It is expected that the multi-step aging treatment, including PA, NA and final AA, would be effective in SiCp/6xxxAl composites to achieve good plastic forming performances and high service strength. Although the effects of PA on AA hardening have been investigated widely in Al-Mg-Si-(Cu) alloys, little attention has been paid to SiCp/6xxxAl composites. SiCp can significantly change the conditions of the Al matrix. For example, interface reactions between SiCp and the Al matrix can deplete Mg and increase the content of Si, resulting in a reduced Mg/ Si ratio [23–26]. Further, the coefficient of thermal expansion of SiCp is  $4.30 \times 10^{-6} \text{ K}^{-1}$ , while that of the Al matrix is  $23.63 \times 10^{-6} \text{ K}^{-1}$  [27]. During quenching, such a huge thermal mismatch can lead to inhomogeneous plastic deformation in the Al matrix, and then results in statistically stored dislocations [28,29]. These dislocations can annihilate vacancies, thereby reducing vacancy concentrations [30]. The aforesaid factors can affect the clustering and precipitation behaviors during aging, for instance, by accelerating the kinetics and changing the features of clusters or precipitates [31-33]. To apply the results of 6xxxAl alloys to SiCp/6xxxAl composites, it is necessary to investigate how these factors affect the PA hardening effect.

In this work, the multi-step aging treatment, including PA, NA and final AA, was conducted in the SiCp/6092Al composite and 6092Al alloy. The aims of this study are to (i) clarify the difference in PA hardening effects between the composite and 6092Al alloy; (ii) elucidate the related mechanisms.

# 2. Experimental procedure

17 vol%SiCp/6092Al composite and 6092Al alloy were fabricated by powder metallurgy (PM) technology, using SiCp (99.5 pct. purity) and 6092Al alloy powders with the nominal composition of Al-1.2Mg-0.6Si-1.0Cu (wt.%). The nominal sizes of SiCp and alloy powders were 7  $\mu$ m and 13  $\mu$ m, respectively.

Fig. 1 shows the schematic diagram of fabrication and heat treatment processes. First, the raw powders were mechanically blended for 6 h with a rate of 50 rpm and a ball to powder ratio of 1:1. Second, the asmixed powders were cold pressed in a cylindrical die under a pressure of 50 MPa. Third, the cold-pressed powders were sintered at 600 °C in a vacuum, and then hot pressed into dense billets.

The PM billets were hot extruded to bars at 450 °C with an extrusion ratio of 16:1 under an extrusion rate of 1 mm/s. Rectangular samples, with sizes of 20 mm (along the extrusion direction)  $\times$  12 mm  $\times$  5 mm, were machined from the extruded bars, solutionized at 540 °C for 3 h, and then water quenched to room temperature (RT). The as-quenched samples were subjected to various aging treatments as shown in Table 1.

Brinell hardness and Vickers hardness were measured using a Testor 1080 hardometer under a load of 250 kg and an FV-700 hardometer under a load of 10 kg, respectively. The samples were ground using 2000# sandpapers before the measurements. At least four hardness values for each sample were acquired to calculate the average value. Conductivity was measured using a D60K conductivity tester. Each sample was measured thrice and the average value was calculated.

Differential scanning calorimetry (DSC) was carried out under an argon atmosphere from RT to 450  $^{\circ}$ C with a heating rate of 5  $^{\circ}$ C/min using the TA-Q1000 system. The baseline of pure Al measured with the

# Table 1

Aging conditions and corresponding nominations.

Sample nominations	Aging conditions
direct AA NA <sub>x</sub> /AA	AA at 170 $^\circ \rm C$ for 6 h immediately after quenching NA at RT then AA at 170 $^\circ \rm C$ for 6 h, where x represents NA time
PA-NA <sub>x</sub> PA-NA <sub>x</sub> /AA	PA then NA <sub>x</sub> PA then NA <sub>x</sub> /AA



Fig. 1. Schematic diagram of powder metallurgy, hot extrusion and heat treatment processes.

same heating process was subtracted.

Microstructure examinations were conducted by optical microscopy (OM) and transmission electron microscopy (TEM, FEI Tecnai F20) equipped with high-angle annular dark-field (HADDF) and X-ray energy dispersive spectroscopy (EDS) detectors. Bright-field (BF), and high-resolution TEM (HRTEM) images were acquired with the electron beam parallel to the  $<001>_{Al}$  zone axis. The TEM foil samples were dimpled and ion-milled at -69 °C  $\sim -71$  °C for the composite, and perforated by twin-jet electro-polishing using the electrolyte consisting of 1/3 HNO<sub>3</sub> in CH<sub>3</sub>OH at about -25 °C with a voltage of 12 V for the 6092Al alloy.

#### 3. Results

## 3.1. Variation of hardness during multi-step aging

Fig. 2 shows the hardness of the composite and 6092Al alloy under various aging conditions. The hardness of the composite was always higher than that of the 6092Al alloy due to the direct (load transfer [34]) and indirect (quenching strain [35]) hardening effects of SiCp.

Dashed and dotted lines in Fig. 2 represented the hardness in the direct AA and  $NA_{2w}/AA$  states, respectively. The  $NA_{2w}/AA$  samples exhibited lower hardness than the direct AA samples both for the composite and 6092Al alloy, indicating that NA exerted negative effects on AA hardening. The PA-NA<sub>2w</sub>/AA samples showed higher hardness than the NA<sub>2w</sub>/AA samples, indicating that PA can enhance the hardening response of the NA<sub>2w</sub>/AA samples. The improvement of hardness by PA in the NA<sub>2w</sub>/AA samples was named as the PA hardening effect in this study.

The PA hardening effect is dependent on the features of clusters formed during PA, such as size, density and composition [16,17]. PA cluster features were complexly affected by PA temperature and PA time due to the diffusion-controlled cluster formation. Therefore, the PA hardening effect varied with PA time and PA temperature. For the composite, the PA-NA<sub>2w</sub>/AA hardness arrived at the highest value when PA was conducted at 170 °C for 15 min, indicating that this PA parameter was optimal.

The PA hardening effect in the composite was stronger than that in

the 6092Al alloy.  $\Delta H$ , defined as the difference in hardness between the PA-NA<sub>2w</sub>/AA and NA<sub>2w</sub>/AA samples, was calculated to quantitatively describe the difference in the PA hardening effect between the composite and 6092Al alloy. Fig. 3 shows the variation of  $\Delta H$  with PA parameters in the composite and 6092Al alloy. The composite had larger  $\Delta H$  than the 6092Al alloy under all PA parameters. The difference in  $\Delta H$  between the composite and 6092Al alloy attained the largest value (6 HB) when PA was conducted at 170 °C for 15 min; hence, this PA parameter was chosen for further analysis.

Fig. 4 shows the variation of hardness with NA time of the PA-NA<sub>x</sub>/ AA samples. The hardness did not change with prolonged NA time in the composite but decreased in the 6092Al alloy. The decrement (7 HB) was nearly equal to the difference in  $\Delta H$  between the composite and 6092Al alloy (6 HB, Fig. 3(a)); hence, the stronger PA hardening effect in the composite (compared to that in the 6092Al alloy) originated mainly from the suppressed negative NA effect. In most of the previous studies, the PA hardening effect was associated mainly with the PA temperature, PA time and PA cluster characteristic [21,36], but this study found that the NA stage after PA was critical.

# 3.2. Microstructures near SiCp-Al interface

The microstructures near SiCp-Al interfaces were critical to understand why PA had a stronger hardening effect in the composite. Fig. 5(a) shows the BF image of dislocations around SiCp. As mentioned above, these dislocations are generated during quenching, and are caused by the mismatch of thermal expansion coefficient of SiCp and the Al matrix [28,29]. The quenching dislocations can improve strength and hardness. The contribution can be approximately calculated by Eq. (1):

$$\Delta \sigma_{dis} = \beta G b \sqrt{\frac{12\sqrt{2}V_{SiC}\Delta CTE\Delta T}{(1-V_{SiC})bd_{SiC}}}$$
(1)

where  $\Delta \sigma_{dis}$  is the contribution of quenching dislocations to yield strength,  $\beta = 1.25$  is a strengthening coefficient, b=0.286 nm is the magnitude of the Burgers vector, G = 26.9 GPa is the shear modulus of aluminum at RT,  $V_{SiC} = 0.17$  and  $d_{SiC} = 7$  µm are the volume fraction and average size of SiCp,  $\Delta CTE$  is the coefficient of thermal expansion



Fig. 2. Hardness of (a, b) SiCp/6092Al composite and (c, d) 6092Al alloy in PA-NA<sub>2w</sub>/AA states under various PA processes: (a, c) PA at 170 °C for various time, (b, d) PA at various temperatures for 15 min.



Fig. 3. Variation of  $\Delta H$  with PA parameters in PA-NA<sub>2w</sub>/AA SiCp/6092Al composite and 6092Al alloy: (a) PA at 170 °C for various times and (b) PA at various temperatures for 15 min.



Fig. 4. Variation of hardness with NA time in  $PA-NA_x/AA$  samples.

mismatch between SiCp and the Al matrix (19.33  $\times$  10<sup>-6</sup> K<sup>-1</sup>), and  $\Delta T$  is the difference between the solution and room temperatures (515 K).

According to Eq. (1),  $\Delta \sigma_{dis}$  (~40 MPa) is mainly related to  $V_{SiC}$ ,  $d_{SiC}$ ,  $\Delta CTE$  and  $\Delta T$ . It is easy to understand that these quenching dislocations will not be affected by NA at RT or PA at 170 °C for such a short time (15 min). Therefore, this study only gives the dislocation distribution in the PA-NA<sub>1h</sub>/AA composite as a representative. And the contribution of quenching dislocations to the hardness under different aging conditions is not discussed in detail.

Fig. 5(a) shows that the dislocation zone extended about 2  $\mu$ m away from the SiCp-Al interface to the Al matrix. Fig. 5(b) shows the schematic diagram of SiCp distribution. The surface distance of neighbor SiCp ( $\lambda$ ) can be calculated by Eq. (2) [37].

$$\lambda = \frac{(1 - V_{SiC}^{1/3})}{V_{SiC}^{1/3}} d_{SiC}$$
(2)

 $\lambda$  was about 6  $\mu$ m in this study, larger than the width of the dislocation zone. Therefore, in addition to the dislocation zones, there also existed the dislocation-free zones in the composite.

Chemical reactions in SiCp-Al interfaces were characterized. As shown in Fig. 6, Mg and O were clearly enriched in the interface. Cu was also enriched, but to a slight degree. However, these elements were uniform in the Al matrix as shown in the linear scanning image. The present phenomenon was consistent with the previous finding that the interface reactions did not affect element distributions in the Al matrix [32]. As the distribution of Mg and O in the SiCp-Al interface was highly relevant, the interface reactions were believed to mainly the oxidation of Mg. Our previous study has confirmed that SiO<sub>2</sub> existed on the surfaces of SiCp [4]. It is well known that Mg and SiO<sub>2</sub> could initiate the following chemical reactions during the fabrication of the composite [23–26]:

$$2Mg + SiO_2 \rightarrow 2MgO + Si \tag{3}$$

$$Mg + 2SiO_2 + 2Al \rightarrow MgAl_2O_4 + 2Si \tag{4}$$

These chemical reactions would deplete Mg and increase the content of Si, such that the Mg/Si atom ratio of the composite was closer to 1 than that of the 6092Al alloy ( $\sim$ 2.5).

# 3.3. AA precipitate observations

#### 3.3.1. Precipitates in the composite

BF images of precipitates were observed in the composite under various aging conditions. Fig. 7(a and b) show precipitates in the NA<sub>2w</sub>/ AA composite. Fig. 8(a and b) and Fig. 9(a and b) show precipitates in the PA-NA<sub>x</sub>/AA (x = 1h and 2w) composite. The precipitates in both the



Fig. 5. (a) BF image of dislocations around SiCp in PA-NA1h/AA composite; (b) schematic diagram of SiCp distribution, and illustration is corresponding OM image.



**Fig. 6.** Typical HADDFimage (left image), corresponding EDS-mapping and linear scanning images (bottom right image) of SiCp/6092Al composite in PA-NA<sub>1h</sub>/AA state. Red line in HADDF image is the linear scanning area. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

dislocation zone (Fig. 7(a) and Fig. 8(a, b)) and dislocation-free zone (Figs. 7(b) and Fig. 9(a, b)) were observed in each composite. Needleshaped precipitates existed in all BF images. These precipitates were elongated along three equivalent  $\langle 001 \rangle_{Al}$ , which was consistent with the typical features of precipitates in 6xxx series alloys [38,39]. The needle-shaped precipitates had two types of cross-sections: dot-shaped and rectangle-shaped (as pointed by arrows). Their structures will be identified by HRTEM in Fig. 10. Dislocations were observed as represented in Figs. 7(a) and 8(a, b), and some precipitates grew along these dislocations.

Figs. 7-9(c, d) show the length distribution and average length of the needle-shaped precipitates. The standard deviation of precipitate length was also calculated to describe numerically the length distribution. The larger the standard deviation, the more discrete the precipitation length distribution. At least 100 precipitates from various images were counted for each sample to ensure the accuracy of the results.

Compared to that in the NA<sub>2w</sub>/AA composite, the precipitate length distribution was more uniform and the average precipitate length was smaller in the PA-NA<sub>x</sub>/AA composite, both in the dislocation and dislocation-free zones. It was found that NA clusters could not act as nucleation sites or precursors of precipitates during AA, leading to the broadened precipitate size distribution and enlarged average precipitate sizes [3,18,19]. The smaller average length and more uniform length distribution of precipitates in the PA-NA<sub>x</sub>/AA composite mean that PA can mitigate the effects of NA clusters on AA precipitates. The reduced precipitate sizes can enhance the Orowan strengthening [40,41], so the hardness of the PA-NA<sub>x</sub>/AA composite was larger than that of the NA<sub>2w</sub>/AA composite.

As shown in Fig. 8, in the dislocation zones, the precipitate length distribution and average precipitate length of the  $PA-NA_{1h}/AA$  composite were similar to those of the  $PA-NA_{2w}/AA$  composite. In the dislocation-free zones (Fig. 9), the  $PA-NA_{2w}/AA$  composite showed



Fig. 7. BF images of precipitates in NA<sub>2w</sub>/AA composite observed in (a) dislocation and (b) dislocation-free zones; (c, d) corresponding results of length measurements of precipitates in Fig. 7(a and b).



Fig. 8. BF images of precipitates in (a) PA-NA<sub>1h</sub>/AA and (b) PA-NA<sub>2w</sub>/AA composite observed in dislocation zone; (c, d) corresponding results of length measurements of precipitates in Fig. 8(a and b).



Fig. 9. BF images of precipitates in (a) PA-NA<sub>1h</sub>/AA and (b) PA-NA<sub>2w</sub>/AA composite observed in dislocation-free zones; (c, d) corresponding results of length measurements of precipitates in Fig. 9(a and b).



Fig. 10. Typical HRTEM images and corresponding FFT patterns of precipitates in Figs. 7–9: (a) β" phases; (b) L phases; (c–d) precipitates along dislocations.

enlarged precipitate sizes compared to the  $PA-NA_{1h}/AA$  composite, but the difference in the precipitate sizes was negligible. Because the precipitates in the  $PA-NA_x/AA$  composite were almost unaffected by NA, the hardness was not changed with prolonged NA time (Fig. 4).

Fig. 10 shows the typical HRTEM and corresponding Fast Fourier Transforms (FFT) images. As shown in Fig. 10(a), the precipitates with

the dot-shaped cross-section had a monoclinic structure with cell parameters of a = 1.516 nm, c = 0.674 nm and  $\beta$  = 105.3°, and were hence determined to be  $\beta$ '' phases (probably Mg<sub>5</sub>Si<sub>6</sub>), the main strengthening phases in Al–Mg–Si-(Cu) alloys [42]. Fig. 10(b) shows that the precipitates with the rectangle-shaped cross-section had a disordered structure and the cross-section was elongated along  $<001>_{Al}$ , which



Fig. 11. BF images of precipitates in 6092Al alloy in (a) NA<sub>2w</sub>/AA, (b) PA-NA<sub>1h</sub>/AA and (c) PA-NA<sub>2w</sub>/AA states; (d–f) corresponding results of length measurements of precipitates in Fig. 11(a–c).

coincided with L phases (uncertain composition but rich in Mg, Si and Cu) [18,43,44].

In the dislocation zones of the composite, two types of precipitates were observed along dislocations. Typical HRTEM and corresponding FFT images are shown in Fig. 10(c and d). Type I had a discrete distribution along dislocations (Fig. 10(c)), while type II was long and curved along dislocations (Fig. 10(d)). Both types of precipitates had no definite unit cells, and corresponding FFT patterns were noisy, indicating that these precipitates were disordered. Similar results were also observed in Al–Mg–Si–Cu alloys with pre-strain during NA [21,45,46], suggesting that quenching dislocations in the composite have the same effects on precipitate structures as deformation dislocations in Al–Mg–Si–Cu alloys. Liu et al. [46] found that the Mg/Si/Cu atom ratio of these precipitates was 4.5/3.4/1, similar to the composition of Q' phases.

## 3.3.2. Precipitates in the 6092Al alloy

The precipitates in the NA<sub>2w</sub>/AA and PA-NA<sub>x</sub>/AA (x = 1h and 2w) 6092Al samples were observed as a comparison to the composite. The measured precipitate length distribution and average precipitate length are shown in Fig. 11. Similar to the composite, the PA-NA<sub>x</sub>/AA 6092Al samples also exhibited smaller average length and more uniform length distribution of precipitates than the NA<sub>2w</sub>/AA 6092Al alloy sample. However, what different from the composite is that the PA-NA<sub>2w</sub>/AA 6092Al sample had broader precipitate length distribution and larger average precipitate length compared to the PA-NA<sub>1h</sub>/AA 6092Al sample, indicating that AA precipitates weakened the Orowan strengthening [40,41], the hardness of the 6092Al alloy in the PA-NA<sub>2w</sub>/AA state was lower than that in the PA-NA<sub>1h</sub>/AA state (Fig. 4).

#### 3.4. Clustering behavior characterizations during NA after PA

#### 3.4.1. DSC analysis

Fig. 12 shows the DSC curves of the composite and 6092Al alloy tested after quenching (AQ), two-week NA (NA $_{2w}$ ), PA at 170  $^\circ$ C for 15 min (PA) and PA-NA<sub>2w</sub>. Exothermic peaks A existed in the AQ samples but disappeared in the  $NA_{2w}$  samples, indicating that peaks A were related to the formation of NA clusters. Moreover, peaks A did not exist in the PA samples, but weaker exothermic peaks B arose at a higher temperature. Peaks B vanished in the PA/NA<sub>2w</sub> samples, suggesting that they were related to the clustering behavior during NA after PA. The existence of peaks B in the PA samples revealed that solute atoms could still form clusters during NA after PA both in the composite and 6092Al alloy, although the clustering behavior was inhibited by precedent PA. Endothermic peaks C were associated with the dissolution of clusters [18,47,48]. These peaks were obvious in the NA samples, but mitigated in the PA and PA/NA<sub>2w</sub> samples, indicating that PA clusters were more stable than NA clusters. Therefore, PA clusters could transform into precipitates during AA.

Peaks D and E were considered as the formation of  $\beta$ '' phases and L phases, respectively [49]. Compared to those in the AQ and NA<sub>2w</sub>

samples, peaks D and E were weaker in the PA and PA-NA<sub>2w</sub> samples, as PA clusters can act as precipitate nucleation sites and thus reduce the heat effects of the formation of  $\beta$ '' and L phases [50]. There also existed some peaks after peaks E, which were considered as the formation of Q phases (peaks F, the equilibrium phases of L phases) and  $\beta$  phases (peaks G, the equilibrium phases of  $\beta$ '' phases) [49,51]. Q and  $\beta$  phases generally arose in the over-aging state. They were not found in TEM observations, indicating that the investigated samples in this study were not over-aged.

## 3.4.2. Hardness and conductivity tests

It was reported that clusters formed during NA, PA and PA-NA<sub>x</sub> only contained a few atoms, and were too small to visualize by TEM [15,52, 53]. Hardness and conductivity tests were common methods for the indirect detection of clustering behaviors [52,54,55].

Fig. 13(a) shows the variation of hardness with NA time for the PA-NA<sub>x</sub> samples. The hardness first reduced and then increased with prolonged NA time in both the composite and 6092Al alloy. However, the composite showed much lower hardness decrement and larger hardness increment compared to the 6092Al alloy. During NA after PA, the formation of clusters leads to the decrease of solute concentration in solution. Cluster formation can increase hardness, while solute reduction can reduce it. The much lower hardness decrement and larger hardness increment in the composite mean that NA clusters of the composite had stronger hardneing ability compared to those of the 6092Al alloy.

Fig. 13(b) shows the variation of conductivity with NA time for the  $PA-NA_x$  samples. Conductivity was sensitive to cluster density [56]. The composite exhibited a much lower reduction in conductivity during NA after PA than the 6092Al alloy, indicating that the composite had much lower increment in cluster density during NA after PA, compared to the 6092Al alloy.

# 4. Discussion

# 4.1. Different clustering behaviors during NA after PA in composite and 6092Al alloy

As shown in DSC results, supersaturated solute atoms still trended to form clusters during NA after PA. The clustering behaviors led to the change in hardness and conductivity. Although grain boundaries and quenching dislocations can also affect hardness and conductivity, it is easy to understand that grain sizes and dislocation density were not changed during NA due to the extremely low temperature (~25 °C). In the composite, the effects of load transfer by SiCp should also be considered. However, the contribution of load transfer to strength was linearly related to the strength of the Al matrix [34,57,58], and the hardness can also be regarded as such [41]. Fig. 13 shows that the variation of hardness and conductivity in the composite was different from that in the 6092Al alloy. It is considered that the difference mainly resulted from the distinct clustering behaviors of the composite and 6092Al alloy, and the contributions of grain boundary, quenching



Fig. 12. DSC curves of (a) SiCp/6092Al composite and (b) 6092Al alloy under various aging conditions.



Fig. 13. Variation of (a) hardness and (b) conductivity with NA time in PA-NA<sub>x</sub> samples.

dislocation and load transfer can be neglected as mentioned above.

Supersaturated solute atoms can form independent clusters during NA or PA [15]. Different from the clustering behavior during NA or PA, supersaturated solute atoms can not only form independent clusters, but also participate into PA clusters during NA after PA [53,59].

As shown in Fig. 13, clusters of the 6092Al alloy exhibited much weaker hardening ability than those of the composite, but led to more significant reduction in conductivity. Therefore, it is inferred that solute atoms in the 6092Al alloy tended to form new clusters during NA after PA.

Because clusters formed during NA, PA or  $PA-NA_x$  are very small in sizes and have no defined structures [53], they can be considered as weak obstacles. As a result, the contribution of these clusters to hardness can be assumed to obey Friedel statistics [40]:

$$H_{cluster} \propto (r_{cluster} f_{cluster}) \tag{6}$$

where  $H_{cluster}$  is the contribution of clusters to hardness,  $r_{cluster}$  is the average radius of clusters, and  $f_{cluster}$  is the volume fraction of clusters. The formation of new clusters could increase  $f_{cluster}$ , but these newly formed clusters were much smaller than PA clusters [53,59], and thus reduced  $r_{cluster}$ . As a result, the hardening ability of these new clusters was weak. However, the formation of new clusters could increase cluster density, which decreased the mean free path of electrons [56,60] and thus reduced conductivity.

The change in hardness and conductivity in the composite was opposite to that in the 6092Al alloy. Clusters formed during NA after PA in the composite had strong hardening ability, but had slight effects on conductivity. It is inferred that solute atoms tended to participate into PA clusters during NA after PA. The incorporation of solute atoms into PA clusters could increase  $r_{cluster}$  and  $f_{cluster}$ , which was beneficial for cluster strengthening. However, this clustering behavior could not increase the density of clusters, so the conductivity was only slightly reduced. The decrease in conductivity may result from the enhanced electron scattering ability of the single cluster due to the increased  $r_{cluster}$ .

## 4.2. Mechanisms for different clustering behaviors during NA after PA

In Al–Mg–Si–Cu alloys, Mg and Si are the main elements to form clusters. Cu can also form clusters, but the clustering ability is much weaker than that of Mg and Si [36]. As mentioned above, the Mg/Si atom ratio of PA clusters is close to 1 (a little larger than 1 in Mg-rich alloys) [15,61]. As the Mg/Si atom ratio of the 6092Al alloy is far larger than 1 (~2.5), Mg will be much surplus after PA. The Mg atom radii is ~12% larger than the Al atom radii and the Si atom radii is ~6% smaller [62]. Hence, the incorporation of Mg into PA clusters will enlarge cluster volume strain. To avoid enlarging volume strain of PA clusters, solute atoms tend to form independent clusters with the assistance of vacancies during NA after PA in the 6092Al alloy. Although Cu (~15% smaller than Al radii [62]) can reduce cluster volume strain,

most PA and NA clusters do not contain Cu due to the weak clustering ability of Cu in the Al matrix [36,63,64].

Vacancies are nucleation sites and diffusion channels for solute atoms [65]. As dislocations can annihilate vacancies, vacancy concentration in the composite is lower than that in the 6092Al alloy. As a result, it is difficult for independent clusters to form during NA after PA in the composite. Dislocations are also diffusion channels for solute atoms. Therefore, it is easy for solute atoms to participate into PA clusters along dislocations during NA in the dislocation zones of the composite. Further, chemical reactions in the SiCp-Al interfaces reduced the Mg/Si atom ratio of the Al matrix (Fig. 6). As a result, it is easier for Mg and Si in the composite to simultaneously incorporate PA clusters during NA, compared to those in the 6092Al alloy.

# 4.3. Effects of different clustering behaviors during NA after PA on AA precipitation and hardness

 $\beta$ '' phases, L phases and disordered phases along dislocations were the main precipitates in this study (Fig. 10). Their Mg/Si atom ratios were all close to 1 [42,46,66]. It is widely accepted that the Mg/Si atom ratio of PA clusters is also close to 1, so it is easy for PA clusters to constitute precipitates during AA [17]. Solute atoms in the composite tend to participate into PA clusters during NA after PA, with the result that the AA precipitate size of the composite was almost unchanged with prolonged NA time, especially in the dislocation zones (Figs. 8 and 9). Although the incorporation of solute atoms during NA may change the composition of PA clusters, this change is slight because the NA clustering behavior is limited by precedent PA.

In contrast, as mentioned above, clusters formed during NA have discrete Mg/Si atom ratios [15]. It is known that most NA clusters cannot transform into precipitates during AA, but trap vacancies, leading to the limited precipitate nucleation and thus larger precipitate size [12,13,16,17]. As it is easy for solute atoms in the 6092Al alloy to form new clusters during NA after PA, AA precipitates of the 6092Al alloy were enlarged with prolonged NA time (Fig. 11).

Due to the effects of the clustering behavior during NA after PA on precipitation being different in the composite and 6092Al alloy, AA hardness of the composite was not affected by NA after PA, while that of the 6092Al alloy was impaired with prolonged NA time. Eventually, PA showed a stronger hardening effect in the composite than that in the 6092Al alloy.

This study also provide inspiration for future researches that in addition to the PA stage, the NA stage after PA should also be heeded when evaluating the PA hardening effect.

# 5. Conclusions

The effects of the multi-step aging treatment, including PA, NA and final AA, on the microstructures and mechanical properties of the SiCp/ 6092Al composite and 6092Al alloy were investigated comprehensively using the hardness test, differential scanning calorimetry, conductivity

test and transmission electron microscopy. The conclusions are summarized below:

- 1. PA could improve the hardness of the NA<sub>x</sub>/AA samples. The composite had a stronger PA hardening effect than the 6092Al alloy.
- Hardness in the PA-NA<sub>x</sub>/AA state was not changed with prolonged NA time in the composite but decreased in the 6092Al alloy; hence, PA showed the stronger hardening effect in the composite.
- 3. The NA stage after PA led to the enlarged AA precipitate size of the 6092Al alloy, but had negligible effects on AA precipitates of the composite. Therefore, NA exhibited different effects on AA hardness in the composite and 6092Al alloy.
- 4. Supersaturated solute atoms tended to participate into PA clusters during NA after PA in the composite, while it was easy for them to form new clusters in the 6092Al alloy. PA clusters could transform into AA precipitates but NA clusters could not; hence, AA precipitates sizes did not change with prolonged NA time in the composite, whereas it was enlarged in the 6092Al alloy.

## CRediT authorship contribution statement

**S.Z. Zhu:** Methodology, Investigation, Visualization, Writing - original draft. **D. Wang:** Data curation, Writing - review & editing, Funding acquisition. **B.L. Xiao:** Conceptualization, Funding acquisition. **Z.Y. Ma:** Supervision, Funding acquisition.

# Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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