Aging behavior of ultrafine grained Al–2 wt%Cu alloy severely deformed by accumulative roll bonding

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Abstract

Aging behavior of an ultrafine grained Al–Cu alloy was studied. Al–2 wt%Cu alloy sheets were solution-treated at 550 °C for 1.8 ks and then severely deformed up to an equivalent strain of 4.8 by the accumulative roll bonding (ARB) process at ambient temperature. The ARB processed material showed the ultrafine lamellar boundary structure with mean boundary interval of 67 nm. The ARB processed sheets and the starting sheets having conventionally coarse grains were aged at 190 °C. The coarse grained specimen for comparison showed typical aging behavior showing in-grain precipitation of the thin-plate shaped θ phase coherent to {001} crystallographic plane of the matrix aluminum. Hardness of the coarse grained specimen increased as precipitation proceeds, reached the maximum around HV60, and decreased owing to over-aging. On the other hand, the ARB processed sample with the ultrafine-grained structure showed very high hardness of HV115 in the as-deformed state, and the hardness decreased monotonously with increasing the aging time. The detailed microstructural observations clarified that recovery and grain growth of the matrix proceeds during 190 °C aging and the precipitation only at grain boundaries occurs in the ultrafine grained material. Instead of the θ phase, the equilibrium θ phase preferentially appeared in the ultrafine grained specimen. The coarsening of the matrix was mainly responsible for monotonous decrease of hardness. The kinetics of precipitation in the ARB processed sample was much faster than that of the coarse grained materials. It was concluded that the aging behaviors in severely deformed material having the ultrafine grained structure are completely different from the conventional ones in the coarse-grained materials.

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1. Introduction

Recently, severe plastic deformation (SPD) of metallic materials has been energetically studied in world-wide scale [1–3]. The SPD above an equivalent strain of approximately 4 can produce the ultrafine grained (UFG) structures whose mean grain size is smaller than 1 μm. Although the minimum mean grain sizes in commercial structural metallic materials have been about 10 μm, the ultra grain-refinement down to nanometer scale is expected to realize superior mechanical properties, such as high strength, high toughness and improved fatigue strength. In fact, the UFG materials obtained by the SPD processes exhibit surprisingly high strength [2,3], which is 2–4 times higher than that of the conventionally coarse-grained materials. Such high strength is explained basically by the strengthening due to grain refinement usually expressed in Hall–Petch equation,

$$\sigma = \sigma_0 + kd^{1/2}$$  \hspace{1cm} (1)

and partly by dislocation strengthening, though the detailed strengthening mechanisms in the SPD materials are still in discussion [4]. However, besides grain refinement strengthening, there are other strengthening mechanisms for metallic materials. Precipitation strengthening is one of those mechanisms, and many commercial alloys, especially aluminum alloys, are strengthened by precipitates. However, aging or precipitation behavior of the UFG materials has been rarely studied until now. Because UFG materials are full of grain boundary [2], it is easily expected that they would show the unique precipitation behaviors which must be curious unknown issues for materials science. One of...
the limited information about this issue is the paper by Murayama et al. [5], in which the microstructural evolution in an Al–3.91 wt%Cu (Al–1.7 at.%Cu) alloy during SPD by equal channel angular pressing (ECAP) [6] was studied. They have reported unusual and interesting precipitation behaviors in aging of the ECAP processed Al–Cu alloy: for example, the formation of metastable phases is skipped in aging of the SPD sample. However, the change in the mechanical properties of the material during aging was not mentioned in the paper. In the present study, the authors aimed to clarify the aging behavior in an ultrafine grained Al–Cu alloy produced by a SPD process. Accumulative roll bonding (ARB) process, which had been originally invented by the present authors [3,7] was used as the SPD process for ultra grain refinement. An Al–2 wt%Cu alloy was selected in this study, in order to simplify the aging behavior. It has been clarified that the temperature increase due to large plastic strain in the SPD processes is significant and the increase in temperature reaches to 130 °C in case of the ARB of 1100 commercial purity aluminum [8]. That is, precipitation may occur even during the SPD processing in certain alloy systems, and it would make the microstructural evolution complicated. However, Silcock et al. [9] showed that the precipitation kinetics in the Al–2Cu alloy is fairly slow and almost no precipitation occurs at 130 °C for aging up to 10 days (864 ks). Namely, precipitation during the ARB processing can be neglected in this alloy. Moreover, it is also reported that only 9h phase precipitates without GP zone at 190 °C in the Al–2Cu alloy [9], which makes the study on the precipitation behavior in the SPD processed UFG material much more clear. In the present investigation, special emphasis was placed on the changes in the microstructures and the mechanical properties during aging after the SPD.

2. Experimental

An Al–2 wt%Cu alloy was made from 99.99 wt%Al and 99.99 wt%Cu by melting and casting method. The ingot of the alloy was homogenized at 520 °C for 24 h, cold-rolled by 93%, and cut into the sheets having dimensions of 2 mm in thickness, 60 mm in width and 150 mm in length. The sheets were solution treated at 550 °C for 1.8 ks and immediately cooled into water. The microstructure was observed from TD of the sheet. Very fine lamellar boundary structure [10] is clearly observed. Such an elongated lamellar structure is the typical ultrafine microstructure observed in the ARB processed materials [3,8,11–13]. It is noteworthy, however, that the present microstructure of Al–2Cu is greatly different from that of the commercial purity aluminum (JIS-1100;
99% Al) ARB processed at room temperature [8]. The lamellar boundaries are quite straight along the rolling direction and there are many dislocations between the lamellar boundaries in the present Al–2Cu alloy (Fig. 2), while the microstructure in the ARB processed 99% Al was rather pancake shaped UFGs nearly free from dislocations inside presumably due to recovery and short range of grain boundary migration enhanced during the process [8]. Extreme fineness of the microstructure in the ARB processed Al–2Cu should be emphasized. The lamellar structure is uniform throughout the sample, and the mean interval of the lamellar boundaries is 67 nm, which is significantly finer than the mean grain thickness (200 nm) of the pancake-shaped UFGs in the 99% Al subjected to an ARB process under the similar conditions [8]. These results indicate that solute atoms greatly affect the microstructure evolution during SPD. That is, solute atoms inhibit recovery during SPD and they are effective to reduce the size of the UFG structures formed through grain subdivision. The SAD pattern in Fig. 2 is not spotty but ring-like, indicating large misorientations exist in the ultrafine microstructure as has been also reported in other kinds of ARB materials [2,3]. The as-ARB processed specimen showed very high hardness, HV 115, which roughly corresponds to flow stress of 380 MPa. This strength is comparable to the tensile strength of some of the commercial 2000 series alloys heat-treated.

Changes in Vickers hardness of the ST specimens and the ARB processed specimens during aging at 190 °C are shown in Fig. 3. The as-ST specimen showed relatively high hardness of HV58. This is probably due to excess vacancies frozen by quenching after solution treatment, because the hardness rapidly recovered down to HV40 during short time aging. Hardness of the ST samples slightly increased with increasing the aging time at the early stage of aging, then significantly increased between $10^4$ and $10^5$ s, showed the maximum value of HV57 at 108 ks (30 h) aging, and then decreased with further increasing the aging period. This completely coincides with the previously reported data in Al–2Cu aged at 190 °C [9] in both hardness level and time dependence. On the other hand, the ARB processed sample showed unique change in hardness. The quite high hardness of the as-ARB specimen rapidly decreased down to HV75 only for 600 s, and it monotonously decreased with increasing the aging time. The ARB specimens did not show the peak of hardness like the ST specimen which is typical in precipitation hardening alloy. The hardness of the ARB specimen after 360 ks (100 h) aging was nearly the same as or rather smaller than that of the ST specimen.

TEM observation showed that no precipitates except for a very small number of those at initial grain boundaries appeared in the ST specimen aged for 1.8 ks (0.5 hr). TEM microstructures of the ST specimen aged at 190 °C for 36 ks (10 h) are shown in Fig. 4. At grain interior (a), thin precipitates aligned along certain crystallographic directions are observed. According to the previous literature [9], these are considered to be $\theta^\prime$ phase. The TEM observations showed that the $\theta^\prime$ precipitates have thin-plate morphology, as is well known [14]. On a grain boundary (b), on the other hand, precipitates whose morphology is significantly different from the in-grain precipitates are seen. Although they aligned along a particular direction possibly owing to the grain boundary structure, their morphology is not thin-plate but
rather massive shapes. The amount of the massive precipitates on the grain boundaries are larger than that of the in-grain precipitates at this stage, demonstrating that the grain boundary is the preferential nucleation site for precipitation as is well known. Fig. 5 shows the TEM microstructures and corresponding SAD patterns of the ST specimens aged at 190 °C for (a) 108 ks (30 h) or (b) 1080 ks (300 h). These figures show the in-grain precipitates. The precipitates are $\theta'$ certainly, since the 110$\theta'$ diffraction spots are clearly recognized in the SAD patterns. In addition to the needle-like contrast along two right-angled directions, weak but clear contrast of the plate-like phases probably included within the thin foil is observed in Fig. 5(a), which confirms that the morphology of the $\theta'$ precipitates is thin plate. The SAD patterns clearly show that they aligned along crystallographic [100] planes of the aluminum matrix. The contrast due to misfit strain at coherent interfaces is recognized in the bright field images. The amount of $\theta'$ precipitates in the 108 ks aged specimen, which corresponds to the maximum hardness in Fig. 3, is much larger than that in the 36 ks aged specimen (Fig. 4). The $\theta'$ precipitates coarsened in the 1080 ks aged specimen, indicating that the decrease in hardness after longer aging period in Fig. 2 is due to over-aging. These are conventionally well-known aging behavior of the present Al–Cu alloy.

Microstructural changes in the ARB specimens during aging were completely different from those in the ST specimens. Fig. 6 summarizes the TEM microstructures of the ARB processed samples aged at 190 °C for various stages of aging.

Fig. 4. TEM microstructures of the solution treated Al–2Cu specimens aged at 190 °C for 36 ks (10 h): (a) at grain interior, (b) around an initial grain boundary.

Fig. 5. TEM microstructures and corresponding SAD patterns of the solution treated Al–2Cu alloy specimens aged at 190 °C for (a) 108 ks (30 h) and (b) 1080 ks (300 h).

Fig. 6. TEM microstructures of the ARB processed Al–2Cu alloy specimens aged at 190 °C for (a) 600 s (10 min), (b) 1.8 ks (0.5 h), (c) 108 ks (30 h), and (d) 1080 ks (300 h). Observed from TD.
periods of time. First of all, the microstructure of the matrix drastically changed from the as-ARB processed one (Fig. 2). The matrix shows no longer the lamellar structure but the somewhat elongated UFG structure only after aging for 600 s at 190 °C (Fig. 6(a)). The grain size (grain thickness) is much larger than the mean interval of the lamellar boundaries in the as-ARB sample (67 nm), and the amount of dislocations inside the UFG grains is much smaller than that in Fig. 2. Furthermore, a number of massive precipitates are observed along the grain boundaries, though almost no precipitates appeared in the ST specimens within such a short period. With increasing the aging time (Fig. 6(b–d)), both the matrix grains and the massive precipitates coarsened, and nearly equiaxed matrix grains free from dislocations inside are seen in the 1080 ks (300 h) aged specimen (Fig. 6 (d)). The TEM microstructures in Fig. 7 obviously show that the precipitates appeared preferentially at the grain boundaries and no crystallographically aligned precipitates with thin-plate morphology were observed at interior of the UFGs. In the 600 s aged specimen, the diffraction spots of both $\theta'$ phase and equilibrium $\theta$ phase are recognized in the SAD pattern (Fig. 7(a)), though the spot of the $\theta'$ is insignificant. Only the diffraction spots from the equilibrium $\theta$ phase are observed in the specimen aged for longer period (Fig. 7(b)). That is, the massive precipitates at the grain boundaries are mostly equilibrium $\theta$ phase. It is noteworthy that the equilibrium $\theta$ phase was not recognized in the ST specimens even after long time aging.

Fig. 8 summarizes the changes in (a) the fraction and (b) the size of the precipitates in the ST and the ARB specimens. The fraction of the precipitates in the ST sample increases with increasing the aging time, and the increasing rate in the fraction decreases after $10^5$ s. On the other hand, the amount of the precipitates in the ARB specimens is much larger than that in the ST specimen at short aging periods. Further, the fraction of the precipitates in the ARB processed specimen has nearly saturated only after aging for 600 s. The amount of the precipitates at later stage of aging is roughly the same in the both ARB and ST samples. It is clearly concluded that the precipitation in the ARB specimens occurs much faster than that in the ST specimens. That is, the UFG microstructure fabricated by the SPD process greatly accelerates the precipitation kinetics in the Al–2Cu alloy. The sizes of the precipitates monotonously increase with increasing aging time in both specimens. Because the $\theta'$ phase precipitated in the ST specimens after aging for 36 ks (10 h) has thin-plate morphology as was shown in Fig. 5, there is large difference between width and length of the precipitates in Fig. 8(b). On the other hand, the aspect ratio of the precipitates in the ARB samples is small, because of their massive morphology (Fig. 6). The sizes of the precipitates in the ARB specimens aged for long periods are much finer than the length of the precipitates in the ST specimens, clearly due to the difference in morphologies.

The mean grain size of the matrix grains in the ARB specimens is shown in Fig. 9. The size along the normal direction of the sheet, in other words the mean thickness of
the grains ($d_i$), is plotted in the figure. The initial interval of the ultrafine lamellar boundaries in the as-ARB specimen is only 67 nm, as was shown before (Fig. 2). It quickly increases up to 300 nm only by holding at 190 °C for a short period (600 s), as was mentioned in Fig. 6. It is interesting that the grain growth seems to be somewhat stabilized until 100 ks, and then the grains greatly coarsened again. This will be discussed in connection with the pinning effect of the precipitates.

4. Discussion

The aging behavior of the SPD (ARB) processed Al–2 wt%Cu alloy sheet having the UFG microstructure was clarified in the present study. It was clearly shown that the precipitation behavior in the SPD/UFG material is completely different from the conventionally solution treated alloy having a coarse grain structure. Precipitation occurred quite quickly in the UFG material. They appear preferentially on the grain boundaries and the morphology of the precipitates is no longer thin-plate aligned along crystallographic {100} plane of the matrix but massive. These results obviously indicate that heterogeneous nucleation of the precipitates at grain boundaries are extremely enhanced in the UFG material. Volume fraction of the grain boundary, $V_{GB}$, has a relationship with area fraction of the grain boundary per unit volume, $S_v$, and thickness of the grain boundary, $t$, as

$$V_{GB} = S_v t$$  \hspace{1cm} (2)

According to quantitative microscopy [15], further, $S_v$ can be related to the three-dimensional mean intercept length of the grains, $L$, as

$$S_v = \frac{2}{L}$$  \hspace{1cm} (3)

for any shapes of the grains. Assuming that the thickness of the grain boundary is 1 nm, as is generally believed, and the mean grain size ($L$) of the matrix in the ST specimens is 50 μm, for example, $S_v$ is $4 \times 10^4 \text{ m}^{-1}$, so that $V_{GB}$ is only $4 \times 10^{-5}$ (0.004%). On the other hand, the ARB processed Al–2Cu specimen had ultrafine lamellar boundary structure whose mean interval is 67 nm (Fig. 2). Providing that all the lamellar boundaries are flat planes parallel to the rolling plane of the sheets, and they are all high-angle grain boundaries, which is not so far from the quantitative evidence in the ARB processed materials [11–13,16–18], $S_v$ for the ARB sample is $1.5 \times 10^7 \text{ m}^{-1}$, so that $V_{GB}$ is 0.015 (1.5%). This value is underestimated, because there are many interconnecting boundaries between the lamellar boundaries [4,13]. Anyway, this is much larger than that in the ST specimens and already not negligible amount for the subsequent microstructure evolution. Furthermore, because the grain boundary is a path for high speed diffusion, the UFG materials have higher diffusivity than the coarse grained materials to result in the acceleration of solid–solid reaction such as the grain boundary precipitation in the present case. That is, the enhanced precipitation (increased kinetics of precipitation) in the UFG material can be qualitatively understood in terms of the drastic increase in the grain boundaries as nucleation sites as well as high-speed diffusion paths in the materials. Additionally, the increase in the number of the point defects like vacancies due to the SPD might be another reason for the accelerated precipitation in the ARB specimen, though it has not yet been proved experimentally.

It is also interesting that the precipitated phase was different between the ST samples and the ARB samples. Only the $\theta^\prime$ phase precipitated in the ST specimens, which agrees with the previous literature, while the equilibrium $\theta$ phase mainly appeared in the ARB specimens. Especially, no in-grain precipitates coherent to the matrix were obtained in the present ARB processed material. Murayama et al. [5] also reported the preferential precipitation of more stable phases in the UFG Al–Cu alloy, skipping the metastable phases, such as GP zone. They made a qualitative explanation that the grain boundary precipitation induces the diffusion of Cu from the grain interior to the grain boundaries, it diminishes the supersaturation of Cu inside the grains, and consequently the driving force for metastable phases decreases. Although the Cu composition in the present alloy is different from their material, similar explanation could stand in the present case as well. It can be concluded, anyway, that the precipitation behavior from the UFG matrix is completely different from that in the coarse grained materials which have been conventionally established.

Another characteristic change in microstructure of the ARB specimen is grain growth of the matrix UFGs. As was shown in Fig. 6(a) and 9, the nanoscale lamellar boundary structure in the as-ARB specimen rapidly changed into the pancake-shaped UFGs only after short time holding (600 s) at low temperature (190 °C). The present authors have
previously studied the annealing behavior of the ARB processed commercial purity aluminum (99% Al), and confirmed that grain boundary migration of the UFGs fabricated by the SPD process actually starts at relatively low temperatures [11,16]. The ARB processed Al–2Cu alloy in the present study had finer structure including larger amount of dislocations than commercial purity aluminum ARB processed under the similar conditions [8,11,16], which is probably the effect of solute atoms. These aspects increase the driving force for recovery, recrystallization and/or grain boundary migration. Therefore, the rapid grain growth in the present material might not be a surprising result. In the present Al–2Cu alloy, however, a number of fine precipitates appear during 190 °C holding simultaneously. These precipitates inhibit grain boundary migration by Zener-drag effect [19]. Actually many precipitates are observed at the grain boundaries in the specimen aged at 190 °C for 600 s (Fig. 7(a)). Here, if the precipitation occurred before the grain boundary migration, isolated arrays of the precipitates should be recognized within the grains. However, most of them locate on the grain boundaries, which suggests that the precipitates appeared after certain amount of grain boundary migration. Actually, the grain growth is once inhibited after 600 s in Fig. 9, which seems to support this consideration. As the specimen is held at 190 °C further, the precipitates coarsen (Fig. 8). Consequently Zener pinning force is weakened, and enhanced grain growth again occurs, as is shown in the longer periods in Fig. 9. That is, the change of the UFG structure is closely related to the precipitation behavior in the alloy systems where low-temperature precipitation can occur.

The strength of the ARB processed specimen monotonously decreased with increasing the aging time. This means that additional hardening by precipitates could not be realized in the present UFG Al–2Cu alloy. This may seem strange since precipitation was significantly enhanced in the ARB material, as was discussed above. However, all the precipitates in the ARB sample were grain boundary precipitates probably incoherent to the matrix, and no coherent phase was observed within the UFGs. As is well known, age-hardening in the Al–Cu system is largely attributed to the coherent strain at interface between the precipitates and the matrix. Furthermore, the size of the massive precipitates in the ARB processed specimens is much larger than the thickness of the thin-plate θ′ precipitates in the ST specimens, and the coarsen with increasing the aging time (Fig. 8). Therefore, it is reasonable that no increase in hardness due to precipitation was observed in the ARB processed material. The Vickers hardness of the ARB processed and aged specimens is plotted as a function of minus square root of the grain size (Hall–Petch plot). This suggests that the in-grain precipitation was observed at temperature much lower than the conventional aging temperature range [20]. Actually, unexpectedly rapid precipitation was also observed in the present UFG Al–2Cu aged at 190 °C. This suggests that the precipitation can be accelerated at much lower temperatures as well, and at lower temperatures which promote larger driving force for precipitation the in-grain precipitation might occur even in the present system. Anyhow, it can be concluded that the precipitation behavior in the UFG materials is dramatically different from that in the conventionally coarse grained microstructure, which should be continuously studied moreover.

5. Conclusions

A solution treated Al–2 wt%Cu alloy was severely deformed by the ARB process to achieve UFG. The aging behavior of the UFG Al–2Cu was clarified. The major results are as follows:

1. The ARB processed material showed ultrathin grained lamellar boundary structure. The mean interval of the lamellar boundaries was 67 nm, which is much finer than those observed in other Al alloys ARB processed. This

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Fig. 10. Vickers hardness of the ARB processed and aged Al–2Cu alloy specimens as a function of minus square root of the grain size (Hall–Petch plot).
indicates that solute atoms significantly retard recovery during the SPD process and they are effective for refinement of microstructure.

2. Precipitation behavior in the ARB processed material having UFG structure was completely different from that in the conventionally coarse grained materials. As is well known in Al–2Cu alloy, only the θ' phase precipitated inside the grains in the coarse grained material. On the contrary, the equilibrium θ phase preferentially appeared in the UFG material. The in-grain precipitates coherent to the matrix were not appeared, but all the precipitates were massive and observed at grain boundaries. The precipitation was greatly accelerated in the UFG material, because of extreme increase of grain boundaries as nucleation sites.

3. The UFG matrix also showed a rapid change at relatively low temperature (190 °C). The ultrafine lamellar boundary structure in the as-ARB processed specimen quickly changed into the pancake shaped UFGs during short time holding at 190 °C. The grain growth of the UFGs was affected by the precipitation.

4. Hardness of the ARB processed Al–2Cu alloy monotonously decreased during aging, and no precipitation hardening was observed owing to the lack of fine and coherent precipitates. Hardness of the ARB processed and aged specimens held the Hall–Petch relationship with the matrix grain size. That is, strength of the present material was mainly governed by grain boundary strengthening.

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