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Microstructure and texture evolution of TRC A8006 alloy by homogenization

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Abstract: The microstructure and texture evolution of twin-roll cast A8006 alloy by homogenization were characterized using scanning and transmission electron microscopy, and the microhardness was tested as well. According to the relationship between dendritic arm spacing and cooling rate the cooling rate of the as-cast twin-roll cast A8006 sheet of 6 mm in thickness was estimated as 1.48×10^3 K·s^{-1}. It is found that the grains and the nanostructural precipitates of the twin-roll cast sheet become coarser after homogenization at 580°C for 4 h in comparison with those after homogenization at 500°C for 8 h. The textures formed after cold rolling and became weaker during homogenization. The increase in hardness of the as-cast twin-roll cast sheets is related to the supersaturated α-Al solid solution and fine microstructure, but the decrease in hardness after homogenization can be attributed to the coarsening of grains and $Al_6Fe(Mn)$ precipitates.

Keywords: aluminum alloys; homogenization; microstructure; textures; hardness

1. Introduction

The rapid solidification process shows a marked enhancement of mechanical properties over the conventionally processed alloys with the same composition through the extension of solid solubility limit, the refinement of microstructure, and the dispersion of secondary phases [1-2]. Rapid solidification is particularly attractive for aluminum alloys because the limited equilibrium solid solubility of some alloying elements in the aluminum lattice can be extended during the rapid solidification process [2-3].

As the good combination of rapid solidification and hot rolling during the fabrication of thin strips or sheets [4-5], the twin-roll casting (TRC) process offers advantages of low capital investment, low operational cost, and the sheets or strips produced having a fine solidification microstructure [6-7]. This process provides the cooling needed for solidification and the rolling necessary for mechanical reduction [8-9].

A8006 (AlFeMn) alloy has been widely used in packaging, microelectronics, architecture, and lithography industries. Although it is well known that the solid solubility of iron in aluminum is very low under equilibrium conditions (0.04wt% Fe) [10-11], the solid solubility can be extended by rapid solidification. Griger *et al.* [11] reported that the iron content increases from 0.041wt% to 0.059wt% when the cooling rate is increased from 1 to 500 K·s^{-1} in Al-0.5wt% Fe samples. Furthermore, during rapid solidification, the cooling rate varies significantly, causing the formation of metastable intermetallic phases (Al_mFe, Al_6Fe, etc.) in addition to the stable Al_3Fe phase [12-13]. However, the formability of Al-Fe alloys mainly depends on the size and distribution of intermetallics, since coarse Al_3Fe eutectic fibers tend to cracks and notches that reduce the formability and fatigue resistance [13]. Usually, residual coarse particles, such as the Fe-rich and Mn-rich phases, deteriorate the extrusion and mechanical performance of Al alloys [14-15]. In addition, the deformation behavior and property of some Al alloys are also influenced by the texture density produced from precipitating of intermetallics [16-17].

Homogenization treatment not only relieves the supersaturation of the matrix by allowing precipitation of secondary intermetallic particles but also changes the primary phase features. Birol [18] reported that a sound homogenization of as-cast TRC Al-Mn strips was identified as the best practice, as the prior cold rolling process did not improve the grain structure or the softening behavior. It is also concluded that a homogenization temperature of at

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least 833 K for TRC Al-1Fe-0.2Si strips must be employed to obtain a coarse particle dispersion which in turn would produce a fine grain size after interannealing [19].

Homogenization is an important process for Al alloys before extrusion and rolling. The present work was undertaken to identify a sound homogenization practice for TRC A8006 sheets. The microstructure and texture of as-cast, cold-rolled, and homogenized sheets of TRC Al-Fe-Mn alloys were characterized, and the homogenization was addressed with an emphasis on the coarsening of intermetallic particles.

2. Experimental procedure

TRC A8006 alloy sheets used for this study were provided by the Technology Strategy Consultants (TSC) Materials Engineering Ltd., UK, and its chemical composition is listed in Table 1. The as-cast TRC sheets of 6 mm in thickness were first rolled into the sheets with the thickness of 3 mm and then were cut into samples with the dimension of 50 mm × 30 mm × 3 mm. Subsequently, these samples were homogenized at 500°C for 8 h and at 580°C for 4 h in an SX-4-10 box-type resistance furnace, respectively.

Table 1. Chemical composition of A8006 alloy used in the present investigation **wt%**

Fe	Mn	Si	Cu	Mg	Cr	Zn	Ti	B	Na	Al
1.487	0.347	0.171	0.06	0.009	0.002	0.004	0.011	$< 1\times10^{-6}$	$< 1\times10^{-6}$	Balance

Microstructures of the as-cast, cold-rolled, and homogenized samples were characterized by a SUPRA55-ZEISS field emission gun scanning electron microscope (FE-SEM) and a Tecnai F30 G2 transmission electron microscope (TEM) equipped with an energy dispersive spectroscope (EDS). The electron back-scatter diffraction (EBSD) technique was used to examine the texture evolution and grain distribution. The samples for FESEM analysis were prepared by conventional metallographic techniques and then polished with colloidal silicon. TEM samples with the diameter of 3 mm were first electro-polished in a solution at −26°C that the volume ratio of ethanol to nitric acid is 7:3, and were then thinned using the twin-jet equipment operated at 30 V. The Vickers microhardness was tested on an HXP-1000M microhardness tester under the load of 0.98 N for 15 s at room temperature, and the microhardness values were the average of six measured results.

3. Results

3.1. Microstructure

Figs. 1(a)-1(d) are the SEM images of TRC A8006 alloy sheets as-cast, cold-rolled, and homogenized at 500°C for 8 h and at 580°C for 4 h, respectively. It can be seen in Fig. 1(a) that the as-cast TRC sheet has a fine homogenous

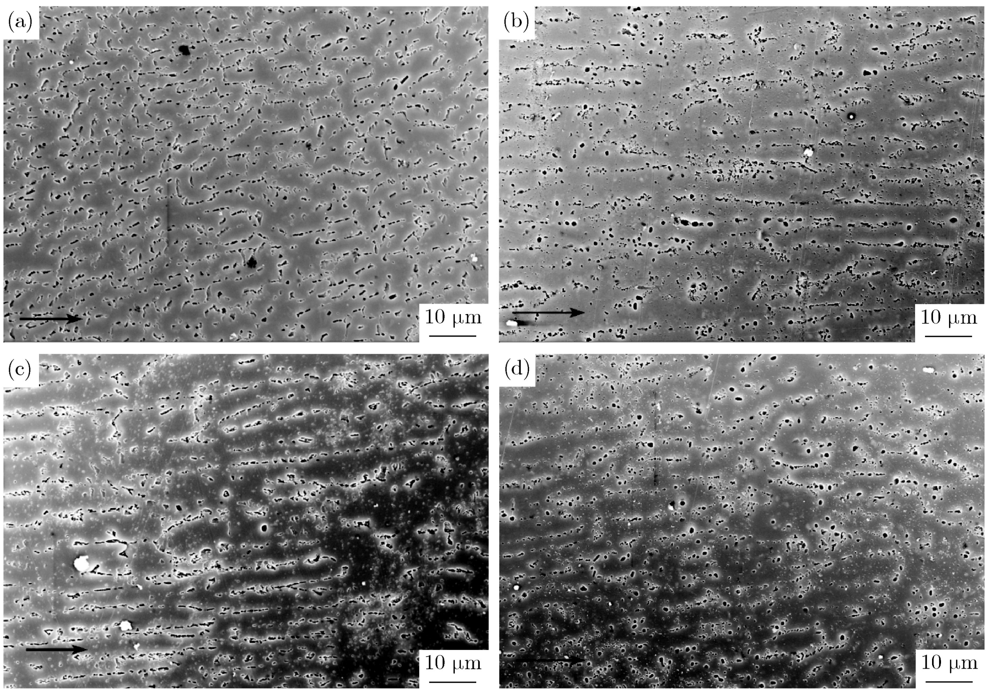

Fig. 1. FESEM morphologies of TRC A8006 alloy with different treatments (the rolling direction is denoted by arrows): (a) as-cast TRC sheet of 6 mm in thickness; (b) cold rolled to 3 mm in thickness; (c) homogenized at 500°C for 8 h; (d) homogenized at 580°C for 4 h.

dendritic structure. The average secondary dendrite arm spacing (SDAS) measured from the FESEM micrograph is about 3.0 μm, whereas the SDAS value of the conventional DC cast alloy reported by Goulart *et al.* [13] is about 19 μm. In addition, fine eutectic crystals are presented on the dendritic boundaries of α-Al dendrites in the as-cast TRC sheet. A single experimental power law for Al-Fe alloys was defined to express the relationship between the secondary dendritic arm spacing λ_2 (μm) and the cooling rate R (K·s^{-1}) [20]: $\lambda_2 = 33.4R^{-0.33}$. Hence, according to the relationship, it is possible to calculate the cooling rate R of 1.48×10^3 K·s^{-1} for the 6 mm-thick TRC A8006 sheet, which is a typical value for the TRC process and is in good accordance with the value reported by Ferry [21].

As seen in Fig. 1(b), the morphology of α-Al dendrites in the cold-rolled sheet is elongated along the rolled direction, while the size of eutectic phase in the homogenized sheets increases with the homogenization temperature rising (Figs. 1(c) and 1(d)).

Figs. 2(a) to 2(d) are the grain morphologies with grain boundaries for TRC A8006 sheets as-cast, cold-rolled, and homogenized at 500°C for 8 h and at 580°C for 4 h, respectively. The grain size of the as-cast TRC sheet with the thickness of 6 mm is about 24.8 μm, whereas the rolled TRC sheet of 3 mm in thickness has a grain size of around 7.6 μm. As shown in Fig. 2(c), the grain size of the TRC sheet after homogenization at 500°C for 8 h is 31.0 μm. However, the grain size of the TRC sheet is 50.0 μm after homogenization at 580°C for 4 h (Fig. 2(d)). It suggests that the increase of homogenization temperature results in the coarsening of grain size and microstructure for TRC A8006 sheets.

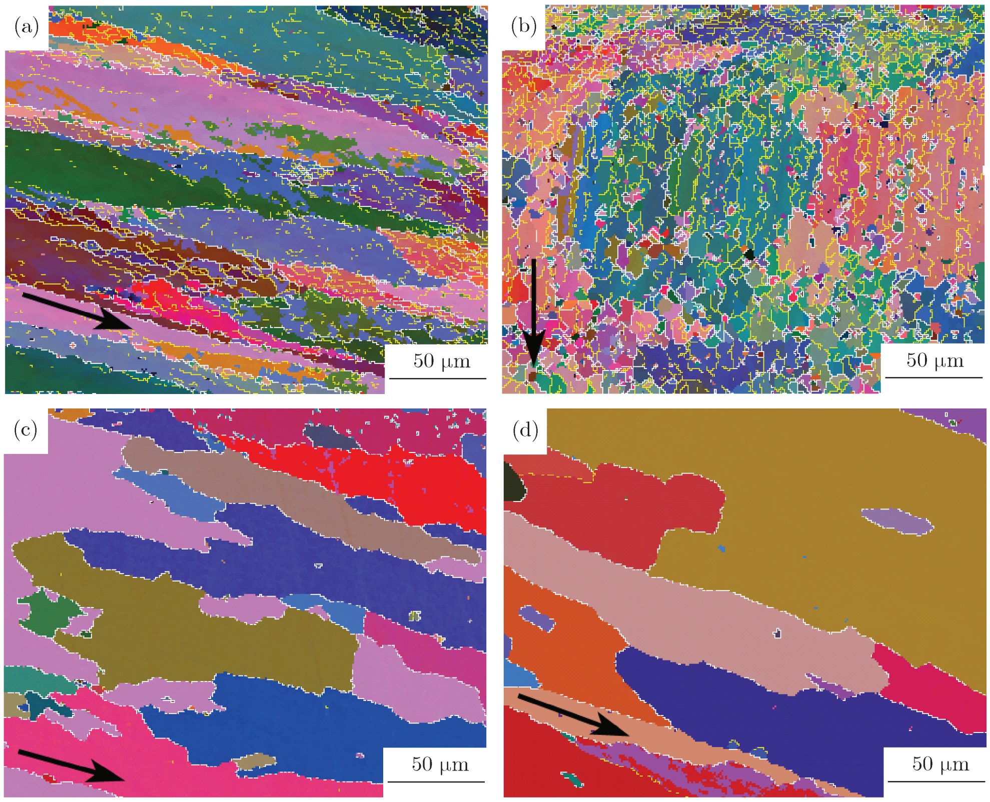

Fig. 2. Grain morphologies with grain boundaries for TRC A8006 alloy with different treatments (the rolling direction is denoted by arrows): (a) as-cast TRC sheet of 6 mm in thickness; (b) cold-rolled to 3 mm in thickness; (c) homogenized at 500°C for 8 h; (d) homogenized at 580°C for 4 h. The white lines are HAGBs (high angle grain boundaries, misorientation > 15°), and the yellow lines are LAGBs (low angle grain boundaries, misorientation < 15°).

3.2. Precipitates

Figs. 3(a)-3(d) are the TEM micrographs of precipitates in TRC A8006 sheets as-cast, cold-rolled, and homogenized at 500°C for 8 h and at 580°C for 4 h, respectively. For the as-cast TRC A8006 sheet, there are rod-like eutectic crystal clusters with the nano-sized diameter of about 110 nm, as shown in Fig. 3(a), while fine dispersive eutectic crystals are present in the rolled TRC sheet, see Fig. 3(b). By homogenization treatment at 500°C for 8 h, the size of rod-like precipitates is in the range of 110-540 nm, while the size of precipitates is in the range of 150-720 nm by homogenization at 580°C for 4 h (Figs. 3(c) and 3(d)). From the electron beam direction $[110]_{\rm Al}$ shown in Fig. 3(e), it can be identified that there is an orthorhombic $Al_6Fe(Mn)$ precipitate in the homogenized sheets, and it has the following structure parameters: space group *Cmcm*, a =0.75518 nm, b = 0.64978 nm, and c = 0.88703 nm. The same result was also reported by Goulart *et al.* [22]. Compared with the morphology of $Al_6Fe(Mn)$ precipitates in the as-cast TRC sheets, the dimensions of precipitates in the sheet after homogenization at 500°C for 8 h increase. For the two homogenized sheets, the size of $Al_6Fe(Mn)$ precipitates further coarsens with the homogenization temperature increasing.

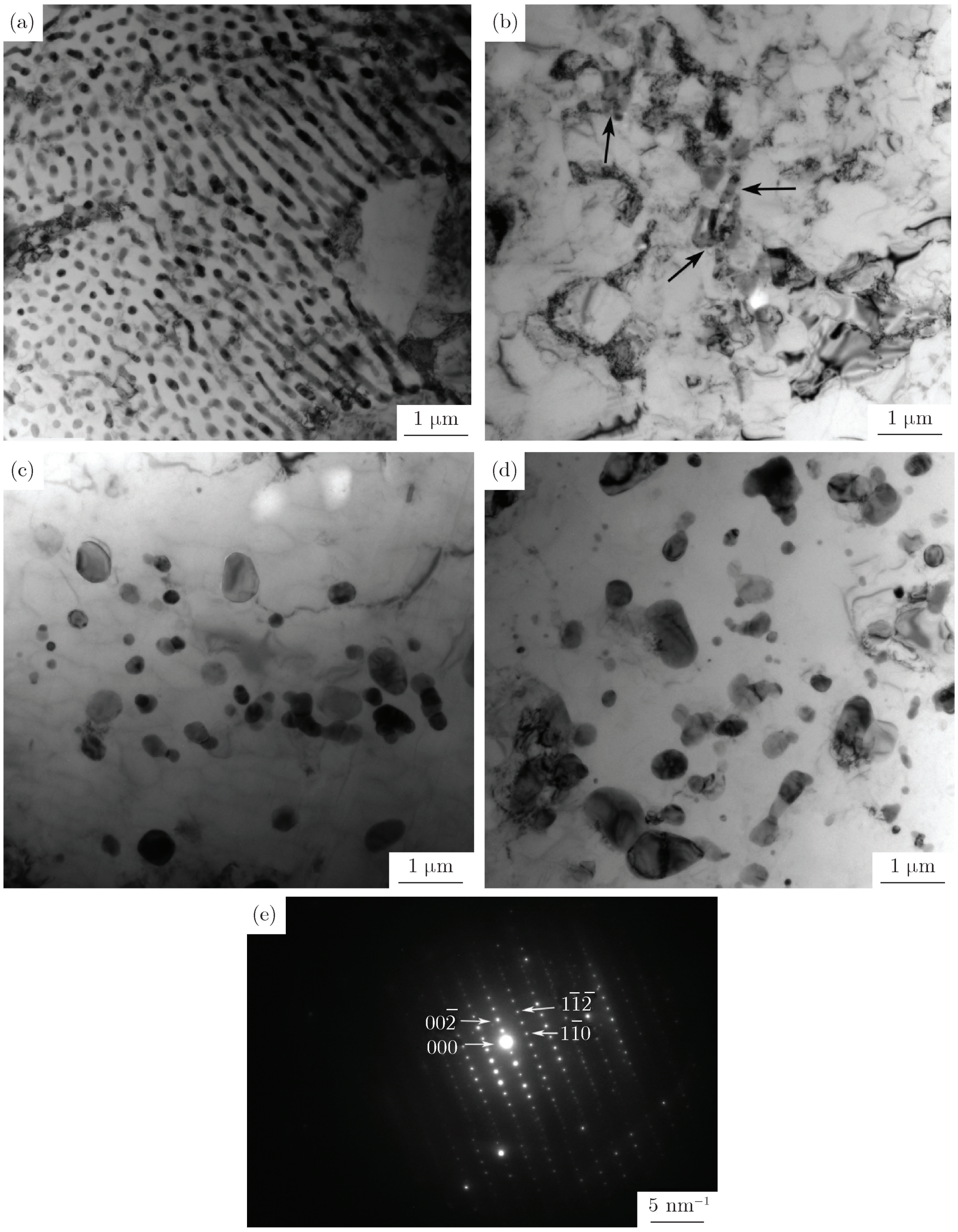

Fig. 3. TEM bright-field images of TRC A8006 alloy with different treatments: (a) as-cast TRC sheet of 6 mm in thickness; (b) cold-rolled to 3 mm in thickness (eutectic crystals are denoted by arrows); (c) homogenized at 500°C for 8 h; (d) homogenized at 580°C for 4 h; (e) SAED pattern viewed along the $[110]_{\rm Al}$ electron beam direction.

3.3. Texture

In Fig. 4, the textures of TRC A8006 sheets after different treatments are displayed by the orientation distribution function (ODF). The results show that the as-cast TRC sheet of 6 mm in thickness has a primary texture {331} <$2\bar{6}1$>, while the texture of the cold-rolled sheet

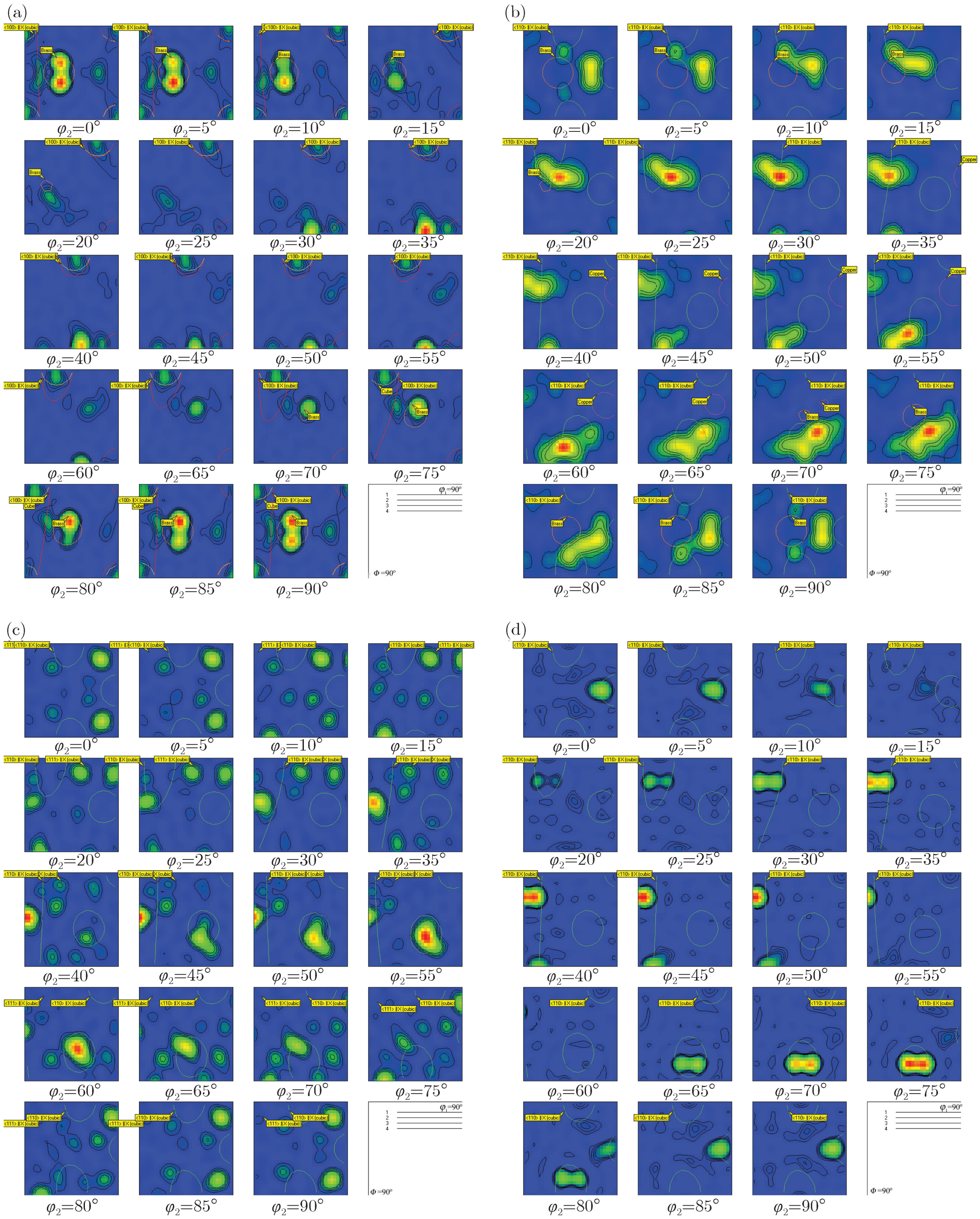

Fig. 4. ODF maps of TRC A8006 alloy with different treatments: (a) as-cast TRC sheet of 6 mm in thickness; (b) cold-rolled to 3 mm in thickness; (c) homogenized at 500°C for 8 h; (d) homogenized at 580°C for 4 h.

presents a typical copper-type texture $\{101\}$ <$21\overline{1}$>. However, it is found that there is inappreciable orientation convergence represented by the red zones in the maps of homogenization samples due to the weak textures, and the deformation textures become weaker when the homogenization temperature increases.

3.4. Microhardness

The Vikers microhardness values of TRC A8006 sheets at different states are given in Table 2. It indicates that the microhardness of the as-cast TRC A8006 sheet is approximately 1.24 times larger than that of the conventionally as-cast Al-3wt% Fe alloy, which is 828.1 MPa [23]. By homogenization at 500°C for 8 h, the hardness of the sheet is distinctly decreased by 68.5% compared with that of the as-cast TRC sheet. Increasing the homogenization temperature to 580°C results in a further reduction in hardness to 698.7 MPa.

Table 2. Microhardness (Hv) of TRC A8006 alloys with different treatments MPa

As-cast	Cold-rolled	Homogenization (500°C, 8 h)	Homogenization (580°C, 4 h)
1025.1 ± 39.2	1205.4 ± 53.9	825.2 ± 47.0	698.7 ± 30.4

4. Discussion

Due to the larger cooling rate (10^3 K·s^{-1}) for the TRC sheet during solidification, the fine dendrite structure was first formed from the supersaturated α-Al solid solution with the presence of undissolved solutes in the Al-matrix, such as Fe, Mn, and Si, and Al-$Al_6Fe(Mn)$ eutectic chains and precipitates distribute along with the dendrite boundaries as shown in Fig. 1. This result was also observed in Refs. [13, 24]. Compared with its conventional casting ingot, the higher microhardness of the as-cast TRC sheet is attributed to the presence of the fine structure and the supersaturated solid solution of α-Al. In addition, for the as-cast TRC sheet, crystallographic texture was formed by the severe plastic deformation of twin-roll casting processing, which has a typical rolling texture of {331} <$2\overline{6}1$> [25].

When the cold-rolled TRC sheet was decreased from 6 to 3 mm in thickness, the presence of large amounts of solutes can cause a strong solute-dislocation interaction and retard the kinetics of both recovery and recrystallization, resulting in the formation of fine grains [26]. The precipitation of fine intermetallics during rapid solidification may also affect the softening behavior of cold-rolled aluminum alloys [27]. Compared with the as-cast TRC sheet, the larger microhardness of the cold-rolled sample could be attributed to the presence of fine grains and higher dislocation density.

During deformation, a typical rolling texture can be generated from strain deformation. Low angle boundaries are formed by dynamic recovery during the deformation. In A8006 alloy with a low solute content, low misorientation boundaries are formed, as shown in Fig. 2(b). These form a cell structure, which tends to remain equiaxed during deformation due to the transient nature of such boundaries [28]. If an assembly of randomly oriented grains is deformed, the orientation spread narrows as the deformation texture develops. In some cases, grain orientations may eventually converge, resulting in the lowering of some boundary misorientations as deformation proceeds. In addition, although the grain size has no significant effect on the deformation texture, local fine precipitate particles arrange the deformed texture by certain grain orientation [28]. Therefore, the typical copper-type texture {101} $< 21\overline{1} >$ was exhibited in the cold-rolled sheet.

Such morphology of precipitation changes by increasing temperature with high growth rates following a power law function of time ($\propto t^{1/3}$), mainly through the volume diffusion mechanism or by structure coarsening [29]. It indicates that a higher homogenization temperature increases the growth rate of $Al_6Fe(Mn)$ precipitate. The reduction in hardness for the homogenized sheet (500°C for 8 h) is related to the coarsening of the microstructure and the disappearance of internal stresses, as well as the precipitating and coarsening of $Al_6Fe(Mn)$ intermetallics [30]. Further reduction in hardness for the homogenized sheet (580°C for 4 h) is due to the coarsening of $Al_6Fe(Mn)$ precipitate particles and the softening of the Al-matrix. Since an increase in homogenization temperature increases the size of the individual precipitate particles, the gradual reduction of solutes results in the gradual softening of the Al-matrix [31]. By homogenization, high angle boundaries are formed by recrystallization and grain growth (in Figs. 2(c) and 2(d)). If an assembly of randomly oriented grains is homogenized, texture and its components in the samples tend to be more homogenized because of the grain rearrangement and cancellation of dislocations during homogenization [16-17]. In addition, coarse precipitate particles weaken the grain orientation. Therefore, the textures become weak after homogenization, and the textures become weaker with the increase in homogenization temperature.

5. Conclusions

(1) Compared with the TRC sheet homogenized at 500°C for 8 h, the grain size and nanostructural $Al_6Fe(Mn)$ precipitates of the TRC sheet homogenized at 580°C for 4 h become coarser.

(2) Cold rolling is beneficial for the formation of textures, but the textures become weak after homogenization. In addition, the increased homogenization temperature can weaken the textures.

(3) The increase in hardness of the as-cast TRC sheet is related to the supersaturated α-Al solid solution and fine microstructure, whereas the decrease in hardness after homogenization can be attributed to the coarsening of grains and $Al_6Fe(Mn)$ particles.

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