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Tensile behavior of 3104 aluminum alloy processed by homogenization and cryogenic treatment

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Abstract: The mechanical properties of 3104 aluminum alloy processed by different combinations of cryogenic and homogenization treatments were studied. The 3104 aluminum alloy processed by the cryogenic treatment followed by homogenization exhibited an enhancement in the tensile strength, yield strength, and elongation by 29%, 41%, and 11%, respectively, as compared with a sample processed by the conventional homogenization treatment. The stress–strain curve of the sample processed by the homogenization treatment exhibited the Portevin–Le Chatelier effect, whereas the sample processed by the cryogenic treatment did not. Further, the cryogenic treatment could accelerate the precipitation of secondary phase particles for the sample processed by a deep cryogenic treatment, followed by a homogenization treatment, which enhanced the dislocation pinning effect of the solvent atoms and thus improved the critical strain.

Key words: deformation; homogenization; cryogenic treatment; microstructure; Portevin–Le Chatelier effect

1 Introduction

The 3104 aluminum alloy has higher strength, superior corrosion resistance, and good deep-drawing and thinning tensile properties; therefore, it has been widely used as food packaging material [1−3]. The tanks used for food packaging are becoming increasingly thinner; thus, there is an increased demand for aluminum materials with comprehensive properties. As a result, many studies have focused on the optimization of hot-rolling and cold-rolling deformation parameters [4], melt treatment [5], and the development of new heat-treatment techniques, such as homogenization and intermediate annealing to precisely regulate and control the microstructure of the alloy. During the casting process, severe dendritic segregation is likely to occur for the 3xxx alloys, and a large number of non-equilibrium eutectic structures may also form, where Al_6Mn, $Al_6(Fe, Mn)$, $Al_{12}(Fe, Mn)_3Si$, etc will exert a significant effect on the deformation, recovery, and recrystallization behavior of the 3xxx alloys [6−8]. Therefore, it is necessary to eliminate or reduce the heterogeneity of the chemical components and structures in order to improve the hot-working plasticity of the ingot via the homogenization treatment.

The cryogenic treatment, an extension of the conventional heat treatment, is a process that places the processed materials into a specific and controllable low-temperature environment, resulting in a change in the microscopic structure of the materials, thereby improving their properties. This treatment has been extensively used for traditional iron and steels [9,10]; titanium- [11], magnesium- [12], and copper-based alloys [13] to enhance the abrasion resistance, rigidity, and service life of the workpieces. Moreover, cryogenic and conventional heat treatments have been employed to produce highly durable vehicle components, as they help convert retained austenite into martensite and promote carbide precipitation [10]. CHEN and LI [14] studied the effect of a cryogenic treatment on commercial A1 alloys and found that the cryogenic treatment could create a α(Al) grain preferred orientation, which is corroborated by XRD results. However, this grain preferred orientation depends on the alloy components and engineering process.

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Many studies have been conducted on the homogenization and annealing parameters affecting the structure and properties of the 3xxx alloys. However, there are few reports on the effects of combined heat treatments such as a cryogenic treatment followed by homogenization. This study focused on the effects and mechanisms of different heat-treatment regimens, e.g., a cryogenic and homogenization treatment, on the mechanical properties of recycled aluminum to provide a reference for developing new process specifications for the heat treatment of recycled 3xxx alloys.

2 Experimental

Recycled 3104 aluminum alloy ingots from scrap aluminum pop cans pre-processed by depainting, fusion, melt treatment, and composition adjustment were used. The ingots were then remelted and refined using C_2Cl_6 and poured into a metal mold preheated at a temperature of 250 °C for the preparation of tensile samples. The chemical compositions of the samples were tested using a direct-reading spectrometer (Spectro MAXx-05; Mn, 0.93%; Mg, 1.14%; Cu, 0.24%; Si, 0.1%; Fe, 0.13%; and Al, 97.46%). The homogenization and cryogenic treatments were performed in an integrated hot-air furnace and liquid nitrogen, respectively. The detailed heat-treatment processes are listed in Table 1. The tensile sample was processed by the heat treatment and fabricated into a standard sample with a diameter and gage length of 5 and 25 mm, respectively.

Table 1 Combined heat treatment regimes for recycled 3104 alloy

Nomenclature	Heat treatment
HT	Homogenization treatment at 580 °C for 10 h
DCT	Deep cryogenic treatment in liquid nitrogen for 24 h
H-DCT	Homogenization treatment at 580 °C for 10 h followed by deep cryogenic treatment in liquid nitrogen for 24 h
DC-HT	Deep cryogenic treatment in liquid nitrogen for 24 h, followed by homogenization treatment at 580 °C for 10 h

Mechanical property tests were conducted using an electronic universal material testing machine (MTS-CMT5105) with an error less than 0.5 MPa. The stretching velocity was 1 mm/min at the corresponding nominal strain rate of 6.7×10^{-4} s^{-1}, and a mechanical property value was calculated as the mean of at least five measurements. Fracture observations were carried out using scanning electron microscopy (SEM; Hitachi SU−1510), and transmission electron microscopy (TEM; JEM−2100F) with energy dispersive spectrometry (EDS; AMETEK) was carried out on foils prepared by polishing by conventional thinning and twin-jet electropolishing using HNO_3 (30% in volume fraction) and CH_3OH (70% in volume fraction) solutions.

3 Results

3.1 Tensile test of 3104 alloy with different heat-treatment regimes

Figure 1 shows the stress–strain relationship at room temperature for a 3104 aluminum alloy sample processed by different heat-treatment processes. It can be observed that the stress–strain curve of the sample processed by the DCT did not exhibit any sawteeth, whereas that processed by the homogenization treatment exhibited sawteeth. During the early stages of strain, the curve is relatively smooth; however, when the strain develops to a certain degree, the stress–strain relationship curve becomes irregular and exhibits sawtooth waves (serrated flow), i.e., the Portevin–Le Chatelier (PLC) effect [15,16]. The critical strain (ε_c), sawtooth amplitude ($\Delta\sigma$), and sawtooth spacing ($\Delta\varepsilon$) are defined in Fig. 1. The critical strain is the strain at which a stress shock appears in the tensile curve. In general, the critical strain refers to the strain in the first sawtooth with an amplitude greater than the experimental error. The sawtooth value is the difference between a stress peak and its next adjacent stress valley, and this value is not always constant during the deformation of the sample. In general, the mean amplitude of the sawteeth within a certain strain is extracted as the characteristic quantity of the PLC effect for analysis. The sawtooth spacing is the difference between the strains of two adjacent peaks. The sample processed by only the first HT (when the strain reaches 3.75%) exhibits a relatively small sawtooth with

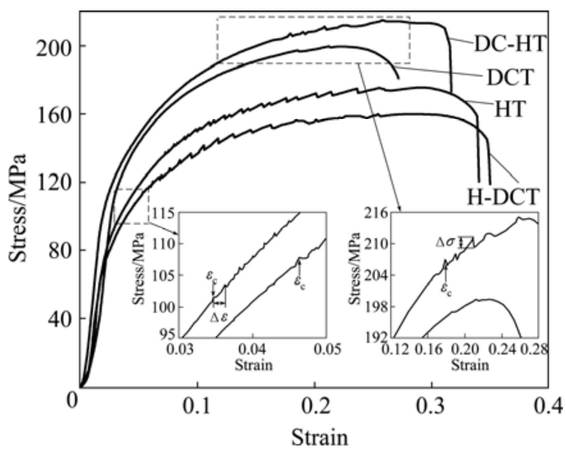

Fig. 1 Stress–strain curve of 3104 alloy with different heat-treatments at strain rate of 6.7×10^{-4} s^{-1} at room temperature (Insets: magnified portion of the curve for defining the critical strain ε_c)

a maximum sawtooth amplitude of approximately 2.7 MPa and a sawtooth spacing of approximately 0.017, whereas the sample processed by the DC-HT exhibits a sawtooth only when the strain reaches 18% with a sawtooth amplitude of approximately 2 MPa and a sawtooth spacing of approximately 0.015. In these three heat-treatment processes (HT, H-DCT, and DC-HT), the sawtooth amplitude gradually increases, then disappears when the strain reaches 27%, and is sustained until necking fracture of the sample.

Figure 2 shows the tensile properties of the samples processed by different heat treatments at room temperature. It can be observed that the sample processed only by the HT exhibited a tensile strength, a yield strength, and an elongation of 165 MPa, 74 MPa, and 18%, respectively. The samples processed by the HT-DCT or HT mainly exhibited consistent tensile and yield strengths. However, the elongation of the HT-DCT sample was 26% higher than that of the HT sample. The elongation of the sample processed by the DCT was the lowest (δ=15%), but its tensile and yield strengths significantly increased to 199 and 107 MPa, respectively, which were 20% and 45% higher than those of the sample processed only by the HT. However, the sample processed by the DC-HT exhibited the highest tensile strength of 213.89 MPa, which is 29% higher than that of the sample processed by the HT. The yield strength of the sample processed by the DC-HT is equivalent to that of the sample processed by only the DCT, but its elongation was 11% higher than that of the sample processed by the HT and 37% higher than that of the sample processed by the DCT treatment. Therefore, the sample processed by the DC-HT possesses superior comprehensive mechanical properties.

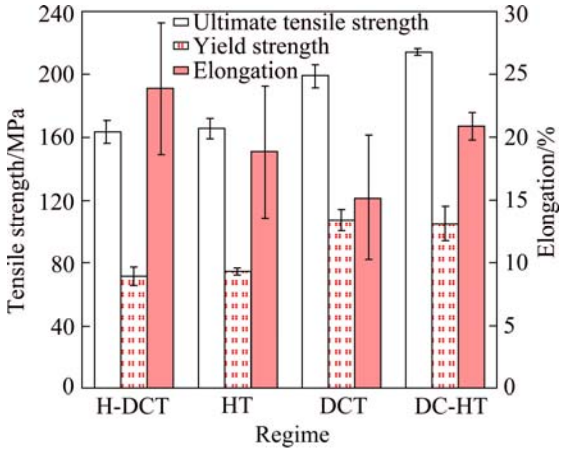

Fig. 2 Effects of different heat-treatment regimes on tensile properties at room temperature

3.2 SEM investigation of fractures of 3104 alloy with different heat-treatment regimes

Figure 3 shows the SEM images of the fracture cross-sections of 3104 aluminum alloy with different heat treatments. A large number of dimples are observed in all samples, which indicates that the tensile fracture mode is typical ductile fracture [17]. It is obvious that the sample processed by the DCT exhibits a larger and shallower dimple, as shown in Fig. 3(a), whereas the samples processed by the HT and H-DCT exhibit many

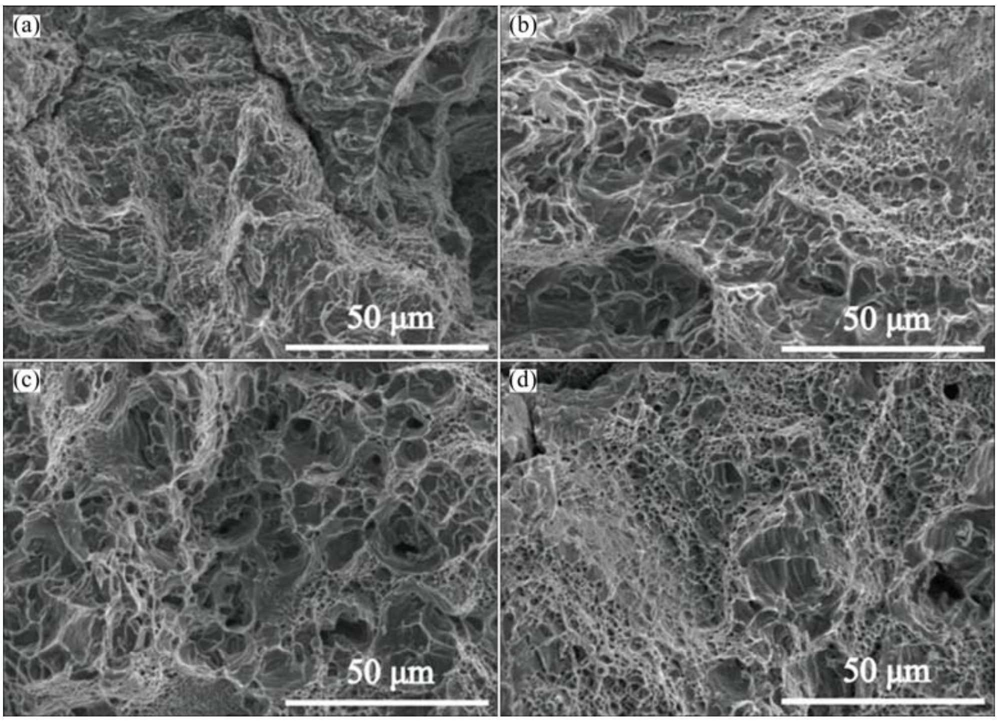

Fig. 3 SEM images of 3104 alloys with different heat-treatment regimes: (a) DCT; (b) HT; (c) H-DCT; (d) DC-HT

smaller and deeper dimples, as shown in Figs. 3(b) and (c). In contrast, the sample processed by the DC-HT exhibits a large number of evenly distributed and smaller dimples, which indicates that it has a good plasticity.

4 Discussion

TEM images of the samples processed by different heat treatments are shown in Fig. 4. According to the EDS analysis (Table 2), the structure of the 3104 aluminum alloy processed by the homogenization treatment at 585 °C exhibits a sheet Al_6Mn phase (labeled as A in Fig. 4(a)). Further, the sample processed by the DC-HT exhibits a granular $Al_{12}Mn_3Si$ phase (labeled as B in Fig. 4(c)) with a size of approximately 0.3 μm, which is considered to be the cubic phase that is coherent or semi-coherent with the aluminum matrix [6]. The precipitations of the samples processed by the DC-HT exhibited a smaller size and more uniform distribution compared with the samples processed by only the homogenization treatment. It can be observed from Figs. 4(b) and (d) that the structure of the sample processed by the cryogenic treatment as the final heat treatment exhibits a large number of dislocations and dislocation tangles (Figs. 4(b) and (d)).

The PLC effect is caused by the interaction between the solute atoms and dislocations. Further, the critical strain is affected by factors such as the composition, the concentration of solute atoms, the diffusion rate, and the heat-treatment state, whereas the sawtooth amplitude is affected by solute diffusion and the blocking time of a dislocation [15]. The samples processed by the homogenization treatment exhibited a large number of precipitated phases, which may block dislocations and offer more time for the solutes to segregate in the dislocations, thereby demonstrating the PLC effect.

The sample structure processed by the H-DCT possessed a large number of dislocations and dislocation tangles. During deformation, the mobile dislocations will be locked by the tree dislocations and precipitated phases. As a result, the pinning effect of the solute atoms is greater, and more time and strain are required to accumulate energy for massively improving the mobile dislocations, leading to increase in the critical strain and sawtooth spacing. However, the sample processed by the DC-HT will produce a very large internal stress in the sample, generating a large number of dislocations and substructures [18], which can further contribute to the formation of densely distributed dislocations or dislocation tangles in the sample. These dislocations or dislocation tangles can serve as the locations for heterogeneous nucleation of the Al_6Mn and $Al_{12}Mn_3Si$ phases and promote the diffusion of atoms such as Si and Mg [7]. As a result, the particles of the precipitated secondary phase are distributed evenly, and the volume fraction of the precipitated phase, where the dislocations

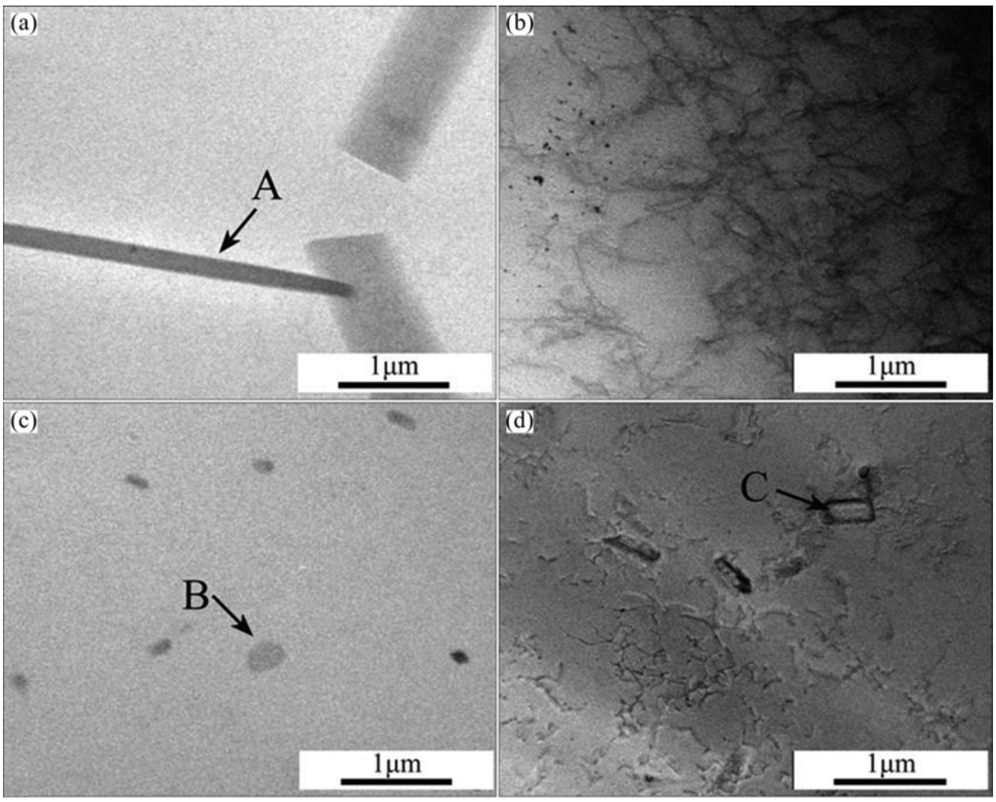

Fig. 4 TEM images of 3104 alloy with different heat treatments: (a) HT; (b) DCT; (c) DC-HT; (d) H-DCT

Table 2 Compositions of precipitation phases marked in Fig. 4

Mark	Elemental composition/%				Nomenclature
	Al	Mn	Fe	Si	
A	86.0	14.0	−	−	Al_6Mn
B	79.2	15.1	−	5.7	$Al_{12}Mn_3Si$
C	76.4	16.0	2.0	5.6	$Al_{12}(Mn, Fe)_3Si$

cannot partition, increases. Notably, the secondary phase particle is not only an obstacle for the dislocation but also a dislocation source. When the secondary phase particle is small and fine, such an action can activate the dislocations around the particles [14], thereby suppressing the coplanar slipping tendency of the alloy while activating cross-slipping, which promotes the uniform deformation of the alloy and improves its toughness and comprehensive mechanical properties.

Although a large number of dislocations appear in the matrix of the sample processed by the DCT, the precipitated phases are still insufficient. During the movement of dislocations, the solvent elements did not have enough time to diffuse and further pin the dislocations; therefore, no sawtooth PLC phenomenon was observed. In summary, the 3104 aluminum alloy processed by the DC-HT exhibited accelerated dispersion and precipitation of the secondary phase, and a large number of fine and evenly distributed precipitated phases enhanced the pinning effect of the solvent atoms on the dislocations [19], which significantly blocked the mobile dislocations. Therefore, the mobile dislocations may abundantly accumulate in front of the precipitated particles, which increases the local stress, sawtooth amplitude, and sawtooth spacing. Therefore, an increase in the amount of the precipitated phase will inevitably lead to a strengthened blocking effect.

5 Conclusions

1) The sample processed by the DC-HT exhibited superior comprehensive mechanical properties including a tensile strength, a yield strength, and an elongation of 214 MPa, 105 MPa, and 20.8% respectively, which are 29%, 41%, and 11% higher than the mechanical properties of the corresponding sample processed by the conventional homogenization treatment.

2) The SEM analyses of the fracture surfaces indicate that the tensile fracture mode of 3104 alloy is typical ductile fracture, whereas the sample processed by the DC-HT exhibits a large number of evenly distributed and smaller dimples, which indicates that it has a good plasticity.

3) The stress–strain curve of the sample processed by the homogenization treatment exhibits the PLC effect, whereas the sample processed by only the DCT shows no such effect. The cryogenic treatment in the DC-HT could accelerate the precipitation and dispersion of secondary phase particles, which enhanced the dislocation pinning effect of solvent atoms and improved ε_c.

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均匀化和深冷处理对 3104 铝合金变形行为的影响

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摘 要：利用电子万能拉伸试验机、扫描电子显微镜(SEM)、透射电子显微镜(TEM)研究 3104 铝合金经不同复合热处理制度(深冷处理+均匀化处理)处理后的力学性能和变形特征。先经深冷处理再均匀化处理的试样具有优异的综合力学性能，其抗拉强度、屈服强度和伸长率分别比仅经常规均匀化处理的试样提高了 29%、41%和 11%。经均匀化处理的试样的应力−应变曲线出现 Portevin−Le Chatelier 效应，而仅经受深冷处理的试样没有出现 Portevin−Le Chatelier 效应。这表明先深冷处理再均匀化处理可以促进 3104 铝合金中第二相弥散析出，大量细小、均匀分布的沉淀相强化了溶质原子对位错的钉扎效应，提高了 3104 铝合金的临界应变值。

关键词：变形；均匀化；深冷处理；显微组织；Portevin−Le Chatelier 效应

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