Investigation on the homogenization annealing treatment of 5052-based aluminum alloys

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Proper homogenization annealing treatment of cast billet is essential to eliminate casting defects and improve the formability of Al alloys. In this work, homogenization annealing treatment in a wide temperature range from 475 to 590°C were conducted on 5052 and Er-modified 5052 Al alloys. The microstructure evolution with annealing temperature was investigated and the homogenized microstructures with and without Er addition were compared and analyzed. The results showed that the optimum homogenization treatment regime is ~560°C for 30 h for both alloys, although the Er-modified alloy can be heat-treated at relatively high temperature than 5052 base alloy. Er addition promotes the dissolution of Al\(\text{Fe}\) phase and helps eliminate the non-equilibrium casting structures. After homogenization, while the needle-liked Al-Fe intermetallic decreases its size from ~20 μm in the as-cast alloy to ~10 μm in the as-homogenized alloy with the stoichiometric ratio of Al to Fe being 4:1, the Al-Fe-Er ternary phase evolves to plate-like particles with the long axis only being ~1 μm. Moreover, Al-Zr-Er ternary granular particles, ~100 nm in diameter, were found to precipitate out from the matrix during the cooling process of the annealing treatment for the Er-containing alloy. It is also revealed that through proper annealing treatment, Er modification helps improve the formability of 5052 alloy.

Keywords: 5052 Al alloy, Homogenization annealing treatment, Cooling rate, Microstructure

Al-Mg series (5XXX) alloys are widely used in various structural components of automotive and transportation industry due to their high specific strength, weldable, good workability and corrosion resistance\(^1\). 5052 Al alloy, containing 2.2~2.8 wt% Mg, has better formability and is more suitable for large wrought products than the other 5XXX series alloys. Demand in light rail train, coal conveyor, refrigerated truck, etc., requires larger-width Al sheets, which in turn, raises requirement of higher formability for Al alloys in order to ensure a homogenous deformation along width direction. Since the wrought Al sheets are hot worked by rolling from a cast billet, the microstructure of the billet plays a crucial role on the formability of the alloy. It is known that proper homogenization annealing treatment of cast billet is essential to eliminate casting defects and improve the formability. Conventionally, 5052 Al alloy billet is heat treated at 450~480°C for 13~36 h\(^2\). Actually, DSC measurement reveals that the initial melting temperature of 5052 Al alloy is above 590°C\(^3\). Higher annealing temperatures can increase the diffusion ability of atoms and improve homogenization efficiency. Moreover, rare earth addition, such as Er, has shown positive effect on alloy plasticity\(^3\~6\), however, the proper homogenization treatment of the Er-containing 5052 Al alloy has not been studied. In this work, homogenization annealing treatment in a wide temperature range from 475 to 590°C were conducted on 5052 alloy, as well as on Er-modified 5052 alloy. The microstructure evolution with annealing temperature was investigated and the homogenized microstructures with and without Er addition were compared and analyzed.

**Experimental Procedure**

The experimental alloys were prepared by mold casting. The actual chemical compositions were analyzed by inductively coupled plasma emission spectrometer (ICP) which revealed a constitution of 2.25% Mg, 0.14% Cr, <0.1 Mn, <0.1 Cu, 0.006% Ti, 0.070% Si, 0.417% Fe, balanced Al for 5052 base alloy and a constitution of 2.27% Mg, 0.10% Cr, <0.1 Mn, <0.1 Cu, 0.020% Ti, 0.081% Si, 0.665% Fe, 0.30% Er, balanced Al for Er-modified 5052 alloy. Homogenization annealing treatment was carried out on both alloys in the temperature range of 475~590°C for 6~30 h. After isothermally holding, one group of

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the billets was cooled with furnace (furnace cooling, FC) and the other group was cooled in open air (air cooling, AC). Samples sectioned from the center of the billets were polished and etched for microstructure observation, by optical microscope and TESCAN VEGA II scanning electron microscope (SEM) equipped with INCA Energy 350 energy dispersive X-ray spectrometer (EDX). The samples were further observed by transmission electron microscopy (TEM, JEM-2010) to study the precipitates. The TEM samples were grinded (Gantan Disc Grinder 623) and then prepared by twin jet electro polishing (Struers TenuPol-2). Microhardness was measured using a Vickers micro-hardness tester with a load of 50 g and duration of 10 s. Cylindrical samples of 56 mm gauge length and 13 mm diameter were used for tensile mechanical testing.

Results and Discussion

Microstructure

The homogenized microstructures of 5052 base alloy and Er-containing alloy were shown in Figs 1 and 2, respectively. With the increase of the isothermally holding temperature, dendrites in the cast microstructure gradually dissolve and dendritic arm spacing gets lager. When annealed at a fix temperature, the dendritic microstructure manifests somewhat finer and homogenous tendency. Moreover, it is noted that, for 5052 base alloy (Fig.1), when the annealing temperature increases to 575°C, the microstructure gets extremely chunky and local re-melting occurs. The situation becomes more pronounced when the temperature further increases to 590°C where extensive re-melting occurs, leading to a typical cast microstructure. It also reveals that cooling manner has an obvious effect on the microstructure, i.e., more homogenous microstructure could be obtained by furnace cooling manner. Comparatively, the effect of cooling manner on the microstructural morphology is more notable at higher temperature. This is largely because while air cooling keeps the cast microstructure obtained at higher treatment temperature, furnace cooling still has enough time to eliminate the cast microstructure during the time span from isothermally holding temperature to room temperature.

The volume fraction of the secondary phases under different heat treatment regimes in 5052 base alloy were measured and listed in Table 1. Except at 590°C, as stated above, the amount of secondary particles is more under furnace cooling than air cooling. This is because two reversal processes should be accounted during the heat treatment, one is the dissolution of the particles into the Al matrix, and the other is the precipitation of solutes from the matrix. Combined with the microstructure in Fig. 1 and the data in

![Fig. 1 — As-homogenized microstructures of 5052 base alloy under different heat treatment regimes](image-url)
Table 1, we can see that 560°C not only favors a fine and homogenous microstructure but also promotes the dilution of the matrix. Since Al-Mg alloys are non-heat treat hardening but derive their strength mainly from solid solution strengthening, the dilution is beneficial to decrease the deformation resistance and working hardening rate of the matrix. From this point of view, 560°C X 20~30 h is the optimum homogenization treatment regime for 5052 base alloy.

For Er-containing 5052 alloy (Fig. 2), the volume fraction of the secondary phases is overall larger than the base alloy, being 1~3% more. Another obvious difference in the as-homogenized microstructure is that the alloy structure is the finest at 575°C and the re-melting temperature also shifts to the higher side, i.e., 590°C. Previous work shows that Er increases the melting point of the α-Al matrix, therefore, the Er-containing alloy can be heat treated at relatively high temperature than the base alloy.

### Phase constitution

To determine the types of the phases after homogenization, SEM and EDX analysis were performed. In both 5052 and Er-modified alloys, the undissolved particles are Fe-containing intermetallic, as illustrated in Fig. 3 whose composition is also inserted in the figure. Compared with the as-cast microstructure, the size of these needle-like particles is reduced and Fe content deviated somewhat from Al$_6$Fe stoichiometry. These brittle intermetallics are harmful to alloy formability. They are normally difficult-to-dissolve phase and still exist in the alloy after homogenization. Therefore, the Fe content should be strictly controlled under a certain limitation.

It should be mentioned that with Er addition, Er is detected in small-sized blocky particle, as illustrated by arrow C in Fig. 3 (b), whose size is just ~1 μm. In cast alloy, Er was always found in Al$_6$Fe phase with an average size of ~20 μm, however, after annealing treatment, only small-sized particles were detected to contain Er. This means that on one side, the Er-doped Al$_6$Fe phase dissolves much easily than Al$_6$Fe phase, and on the other side, Er tends to escape away first from the intermetallics and dissolves into the matrix during the homogenization. Therefore, it is safe to say that Er promotes the dissolution of Al$_6$Fe phase and helps eliminate the non-equilibrium casting structures.
TEM observation

To study the fine precipitates which might form during the homogenization treatment and the possible effects of Er on the precipitation, TEM observation was conducted on samples annealed at 560°C for 30 h with furnace cooling for the Er-modified alloy. Needle-like Al-Fe intermetallics with ~10 μm long in size were frequently identified and the atomic ratio of Al to Fe was measured to be 4:1, as shown in Fig. 4(a,b) and the inserted EDX analysis results. Besides, Al-Fe-Er ternary phase was also observed (Fig. 4c) whose morphology is more like a plate under TEM with the atomic ratio of Al to Fe to Er being approximately 9:2:1. The size of the long axis is ~ 1 μm, which accords with the SEM observation results.

Detailed TEM observation also shows that apart from the Al-Fe-(Er) intermetallic compounds, an Al-Zr-Er ternary granular precipitates, ~100 nm in diameter, was detected with almost a fix Al:Zr atomic ratio of 5:1, as shown in Fig. 5(b) and the corresponding EDX analysis results. These particles are believed to precipitate out during cooling from high isothermally holding temperature to room temperature. The fine precipitates could provide a strong pinning effect on grain boundary motion and thus is beneficial to a fine-grained recrystallization structure through the subsequent hot deformation processing. Besides, short-bar Al-Mg phases with minor Si or Er were occasionally observed (Fig. 5a). These Al-Mg phases are normally several hundred...
nano-meter long and are not thought to precipitate from the matrix during the cooling process, instead, they are most probably excessive phases remained in the as-homogenized structures. Because of their small size and amount, they are not easily observed under OM and SEM. These excessive phases have negligible influence on the alloy workability.

**Mechanical testing**

Microhardness testing shows that annealing temperature and cooling manner both have remarkable influences on the HV values. The testing results for the base alloy and Er-modified alloy annealed at different temperatures for 30 h are given in Fig. 6. With the increase of the annealing temperature, the HV decreases in the whole and reaches its lowest value at 560°C. In addition, alloys cooled with furnace show lower hardness values than alloys cooled in air. These changing tendencies accord well with the microstructure observation. Normally, lower hardness means weaker deformation resistance which is beneficial to subsequent plastic deformation processing. It is noted that the lowest HV value is achieved in Er-containing alloy annealed at 560°C for 30 h. This indicates that precipitation is more sufficient under this regime for the Er-containing alloy and that Er modification helps improve the formability of 5052 alloy.

To further clarify this point, tensile mechanical testing was conducted on alloys with and without Er addition, both annealed at 560°C for 30 h. It is revealed from Fig. 7 that the tensile strength decreases a bit while the elongation is obviously increased with Er addition, indicating improved alloy plasticity in Er-containing alloy.
Conclusions

The following conclusions can be drawn from this study:

(i) The Er-modified alloy can be heat-treated at relatively high temperature than 5052 base alloy. The optimum homogenization treatment regime is ~560ºC for 30 h. The temperature should not exceed 575ºC and 590ºC for the base alloy and the Er-containing alloy, respectively, so as to avoid re-melting.

(ii) After annealing treatment, the size of the needle-liked Al-Fe intermetallic is much reduced in both alloys. For Er-containing alloy, the size decreases from ~20 μm in cast alloy to ~10 μm; the stoichiometric ratio of Al to Fe is 4:1.

(iii) Al-Fe-Er ternary phase evolves to plate-like particles after annealing treatment with the long axis being ~1 μm and the atomic ratio of Al to Fe to Er being approximately 9:2:1. Al-Zr-Er ternary granular particles, ~100 nm in diameter, precipitate out from the matrix during the cooling process of the homogenization treatment for the Er-containing alloy.

(iv) Er promotes the dissolution of Al₆Fe phase and helps eliminate the non-equilibrium casting structures.

(v) The lowest HV value is achieved in Er-containing alloy annealed at 560 ºC for 30 h with furnace cooling. Tensile elongation is much improved by Er addition. Er modification helps improve the formability of 5052 Al alloy.

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