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Effects of cold rolling and heat treatment on microstructure and mechanical properties of AA 5052 aluminum alloy

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Abstract: The microstructures and mechanical properties of homogenized-rolled AA5052 aluminum alloys with different rolling reductions and following annealing treatments were investigated by optical microscope, scanning electron microscope, X-ray diffractometer, micro-hardness and tensile tests. The results show that with increasing rolling reduction, the equiaxed grains are elongated along the rolling direction obviously, and accumulation of rolling reduction increases the work hardening effect, which results in the enhanced strength and degraded plasticity. When rolling reduction is 87%, the ultimate tensile strength reaches 325 MPa but elongation is only 2.5%. There are much more secondary phase precipitates after annealing treatment. With an increase of annealing temperature, the amount of precipitates increases and work hardening diminishes continuously. The elongation is improved to \sim 23% but the tensile strength is decreased to 212 MPa after annealing at 300 °C for 4 h, which are comparable to those of as-homogenized alloy.

Key words: AA5052 aluminum alloy; cold rolling; annealing; microstructure; mechanical properties

1 Introduction

In recent years, aluminum alloys have attracted much more attention due to the fact that they possess light mass, high strength, easy recycling and high corrosion resistance. Because of such attractive features, they have been showing a remarkable increase in the applications in automotive industry and other engineering areas, such as aerospace industry and telecommunication [1–9]. However, the widespread applications of wide aluminum alloy sheets are restricted because of their various and complex process technologies, and higher cost than steel sheets [10–21].

Cold rolling is an important process of wide aluminum alloy sheets. Rolling reduction and the following annealing temperature are two important parameters during the rolling process. The following annealing heat treatment can improve the forming properties. Therefore, it is very necessary to study the effects of rolling reduction and annealing temperature on microstructure and mechanical properties of aluminum alloys [22-32].

AA 5052 aluminum alloy is generally classified as non-heat treatable alloy, and its nominal chemical composition consists of Mg, Fe, Si, Cu, Mn and other elements. In AA 5052 aluminum alloy, the main addition element is Mg, with the content ranging from 2.2% to 2.8%, which strengthens the alloy by forming solid solution [12-18]. A few of researches have been carried out on enhancing mechanical properties of AA 5052 aluminum alloy to meet the needs of modern sheet production. LIU and MORRIS [8] found that the recrystallized grains of AA 5052 alloy could be refined by cold rolling prior to annealing, which was beneficial for improving its mechanical properties. SONG et al [14] studied the mechanical properties of ultrafine grained AA 5052 aluminum alloy produced by accumulative roll-bonding and cryogenic rolling. Their results indicated that the strength of AA 5052 aluminum alloy increased with an increasing number of cycles by accumulative roll bonding processing at 300 °C.

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However, there is limited systematic research work performed to investigate the effects of rolling reduction and annealing on as-rolled AA 5052 aluminum alloy.

The microstructure and mechanical properties of cold-rolled AA 5052 aluminum alloys subjected to different rolling reductions and processed by the subsequent annealing heat treatments were investigated systematically in this work. The corresponding mechanisms on the variation of tensile properties with microstructure were discussed. Our study provides an important basis for improving the mechanical properties and thus developing high performance aluminum alloy sheets for engineering applications.

2 Experimental

The experiment material AA 5052 aluminum alloy (as-cast) was supplied by Southwest Aluminum (Group) Co., Ltd. The compositions of the ingots were analyzed by photoelectricity spectrum analyzer (ARL4460) and the results are listed in Table 1.

Table 1 Chemical composition of AA 5052 aluminum alloy(mass fraction, %)

Mg	Fe	Si	Cr	Mn	Cu	Al
2.7021	0.3614	0.4262	0.16	0.0706	0.0399	Bal.

The as-cast AA 5052 aluminum alloy was homogenized at 470 °C for 15 h and cooled in the air, and homogenized samples were machined into 85 mm \times 50 mm \times 8 mm rectangular as the as-rolled specimens. Then, sheet was rolled in air. The rolling reduction is 15% in every pass. The thickness of sheet was reduced by rolling from 8 to 6.8, 5.4, 4.3, 3.2, 2 and 1 mm, and total rolling reductions are 15%, 33%, 46%, 60%, 75%, and 87%, respectively. The as-rolled alloy with the reduction of 75% was chosen to study the effect of annealing temperature on microstructure and mechanical properties of as-rolled aluminum alloys. Five types of heat treatments were performed on the as-rolled alloy, namely at 220, 250, 300, 350 and 380 °C for 4 h, respectively. As-rolled and following annealing specimens for microstructural observations were conducted on the midsections parallel to rolling direction (RD). They were prepared by mechanical grinding, polishing, and subsequent electrolytic polishing. Electrolytic polishing was carried out in a solution of 20% HClO₄ + 80% C₂H₅OH (volume fraction) for 8–15 s at the voltage of 18 V and the current of 0.7-1.0 A. Microstructures of these alloys were investigated with an optical microscope (OM) and a scanning electron microscope (SEM, TESCANVEGA II LMU) by an accelerating voltage of 20 kV. Analysis of the second phase composition was carried out by energy dispersive spectroscopy (EDS). The grain size of the experimental alloys was counted by an image-pro-plus software. Phase analyses were performed with a Rigaku D/MAX-2500PC X-ray diffractometer (XRD) with Cu K_{α} X-ray source and 2θ range from 10° to 90° at a scanning rate of 0.03 (°)/s. Specimens for hardness tests were prepared by mechanical grinding and polishing, then conducted on a Vickers hardness testing machine with a load of 50 g and a loading duration of 15 s. For each specimen, at least ten indents were performed. Tensile specimens of 5 mm in gauge diameter and 30 mm in gauge length were cut along rolling direction. Tensile tests were carried out by a CMT5105 material test machine with a strain rate of $1 \times 10^{-3} \text{ s}^{-1}$ at room temperature. The 0.2% yield strength (YS), ultimate tensile strength (UTS) and elongation to failure were averaged over three specimens.

3 Results and discussion

3.1 Microstructure characterization

The optical microstructures of as-cast and homogenized AA 5052 aluminum alloys are shown in Fig. 1. The as-cast alloys have typical dendritic structure with α (Al) matrix and a large amount of second phases distributed along the dendritic boundaries. The grain sizes of as-cast and as-homogenized AA 5052 aluminum alloys are 118 and 136 µm, respectively. The grains grow obviously after homogenized treatment. It can also be seen that some large second-phase particles and most of the dendrites are dissolved and disappear in the as-homogenized alloy from Figs. 1(c) and (d).

Figure 2 presents the optical microstructures of cold-rolled AA 5052 aluminum alloys subjected to different rolling reductions. As can be seen from Fig. 2, these samples show typical strained structure, and the grains are severely elongated along the rolling direction for all the samples. The grain sizes of as-rolled alloy with different reductions of 15%, 33%, 46%, 60%, 75% and 87% are 148, 156, 180, 467, 701 and 732 μ m in length, respectively. For the samples with reductions of 15% and 33%, the grains show little change as compared with that of the as-homogenized sample with length of 136 μ m.

However, the grains are elongated along the rolling direction obviously with rolling reduction from 60% to 87%, especially for 87%. The grain size is as large as 732 μ m. When the rolling reduction increases up to 46%–87%, grain boundaries become much coarser and the grains are elongated evidently, especially samples with reductions of 75% and 87%, as shown in Figs. 2(e) and (f).

The XRD patterns of AA 5052 aluminum alloy in different conditions are shown in Fig. 3. As can be seen from Fig. 3, the as-homogenized AA 5052 aluminum alloy consists of α (Al), Mg₂Si and Al₃Fe. However, the



Fig. 1 Optical micrographs of as-cast and homogenized AA 5052 aluminum alloys: (a,b) As-cast; (c,d) Homogenized



Fig. 2 Optical micrographs showing microstructure of cold-rolled AA 5052 aluminum alloys with different reductions: (a)15%; (b) 33%; (c) 46%; (d) 60%; (e) 75%; (f) 87%



Fig. 3 XRD patterns of as-homogenized and as-rolled AA 5052 Al alloy with different reductions

as-rolled AA 5052 aluminum alloys with different reductions are all composed of α (Al), Mg₂Si and Al₃Fe, and no other phases can be detected. This means that this kind of phases of AA 5052 aluminum alloy are unchanged after rolling. While the diffraction peaks are enhanced with increasing rolling reduction, especially for 87% reduction. This implies that with increasing rolling reduction, and the amount of secondary phase increases. The results are consistent with the OM microstructural observations in Fig. 2.

Figure 4 shows the EDS results of as-rolled AA 5052 aluminum alloy with different reductions. In Fig. 4(a), there is a large amount of Mg and Si as well as a few of Fe in Point B; however, Point A only consists



Desition -	x/%					
Position	Al	Mg	Si	Fe		
Α	96.95	3.05	-	_		
В	48.34	25.23	24.69	1.74		
С	78.10	0.91	-	20.99		
D	97.10	2.90	-	_		
Ε	81.12	0.65	-	18.24		
F	34.73	20.54	44.72	_		

Fig. 4 EDS results of as-rolled AA 5052 Al alloy with different reductions: (a) 33%; (b) 75%

of Al and Mg, the Point *C* is much rich in Al and Fe. Combined with Fig. 3, Point *A* should be α (Al) matrix, Points *B* and *C* should be Mg₂Si and Al₃Fe, respectively. In Fig. 4(b), most of Mg exists in the form of solid solution in the matrix. However, the lath-liked dendrites have been broken by the large rolling deformation. Point *D* is rich in Al and Mg, Point *E* mainly consists of Al and Fe, and Point *F* is the mixture of Al, Mg and Si. Combined with Fig. 3, Point *D* may be the α (Al) matrix, Points *E* and *F* are identified as Al₃Fe and Mg₂Si, respectively.

Figure 5 illustrates the microstructures of as-rolled AA 5052 aluminum alloy with 75% reduction under different heat treatments. In Fig. 5, it can be seen that annealing treatments do not change the grain morphology, but the grain size of the alloy is larger with the increase of annealing temperature, especially in Fig. 5(f). The grain sizes of Figs. 5(a)-(f) are 424, 432, 444, 480, 496 and 516 µm, respectively. The grains grow seriously after annealing at 300 °C. Numerous intermetallic particles precipitate from the matrix during annealing and most of these precipitates are distributed along with grain boundaries after annealing treatment. It is found that the precipitates of the alloy are the most when annealing at 250 °C. According to PAN and ZHANG [33] and QIAN [34], there are Al₃Mg₂ precipitates when annealing at 250 °C. In order to observe the change of secondary phase, further SEM and XRD studies were conducted on the annealing samples and the results are shown in Figs. 6 and 7, respectively.

Figure 6 shows the SEM images of as-rolled AA 5052 aluminum alloy with 75% rolling reduction subjected to different annealing heat treatments. It can be seen from Fig. 6 that, the grain boundaries are coarser after annealing, especially in Fig. 6(e), annealing at 350 °C for 4 h. Combined with Figs. 5 and 6, there is elongated fiber microstructure along the rolling direction as the annealing temperature is lower than 300 °C, and the elongated fiber microstructure turns to equiaxed grains above 300 °C. It can be deduced that there is recrystallization at 300 °C. YI et al [35,36] also found that the recrystallization occurs at 300 °C. The following hardness and tensile tests in Figs. 8 and 9 also confirm that.

The XRD patterns of as-rolled AA 5052 alloy with 75% rolling reduction subjected to different heat treatments are shown in Fig. 7. Figure 7(a) shows that as-rolled AA 5052 alloy with 75% reduction is annealed at 250 °C for 4 h, Fig. 7(b) shows that the sample is annealed at 300 °C and Fig. 7(c) shows that the sample is the as-rolled AA 5052 Al alloy with reduction of 75%. As can be seen from Fig. 7, Mg₂Al₃ phase can be detected obviously after annealing. With increasing Mg



Fig. 5 Optical micrographs of as-rolled AA 5052 aluminum alloy with 75% rolling reduction subjected to different annealing heat treatments: (a) Rolled; (b) 220 °C, 4 h; (c) 250 °C, 4 h; (d) 300 °C, 4 h; (e) 350 °C, 4 h; (f) 380 °C, 4 h



Fig. 6 SEM images of as-rolled AA 5052 aluminum alloy with 75% rolling reduction subjected to different annealing heat treatments: (a) Rolled; (b) 220 °C, 4 h; (c) 250 °C, 4 h; (d) 300 °C, 4 h; (e) 350 °C, 4 h



Fig. 7 XRD patterns of as-rolled AA 5052 Al alloy with 75% reduction subjected to different heat treatments

content, the phases are composed of α (Al) and Mg₂Al₃ [1,2,5–10]. All the annealed samples comprise α (Al), Mg₂Si, Al₃Fe and Mg₂Al₃ phases. While the as-rolled sample only consists of α (Al), Mg₂Si, Al₃Fe and no Mg₂Al₃ phase can be detected. Comparing the phase compositions of as-rolled and as-annealed samples, it is known that Mg₂Al₃ phase is induced by annealing heat treatments.

3.2 Mechanical properties

The hardness curves of AA 5052 aluminum alloy under different conditions are shown in Fig. 8. As shown in Fig. 8(a), the hardness of the alloy after the homogenized treatment decreases from HV 61 to HV 57. While it increases rapidly with increasing rolling reduction. This means that there is obvious work hardening during rolling. However, the hardness changes a little from HV 97 to HV 99, when the rolling reduction increases from 75% to 87%, respectively. As the rolling reduction is 87%, the hardness is close to HV 100 that is almost two times that of the as-homogenized AA 5052 aluminum alloy, indicating that the hardness can be improved by rolling. The reason is that with the increase of rolling reduction, the dislocation density increases, so the hardness increases. However, the hardness of the as-cast AA 5052 aluminum sample is higher than that of the homogenized sample and the standard deviation is higher. For as-cast alloy, the microstructure is heterogeneous and there is some segregation, therefore, the deviation is higher. For the homogenized alloy, the microstructure is homogenous, some second-phase particles and most of the dendrites are dissolved or disappear, and the segregation is reduced, so the hardness is lower and the deviation is lower compared with the as-cast alloy. In Fig. 8(b), the hardness decreases notably after annealing. With increasing annealing temperature, the hardness declines. It decreases slightly after

annealing at first, but decreases rapidly after annealing at 250 °C for 4 h. Therefore, it can be deduced that there is recovery and recrystallization during annealing. As we all known, the hardness decreases slightly during the recovery; however, it decreases obviously during recrystallization [37]. This can be seen clearly from Fig. 8(b), after annealing at 220 °C and 250 °C for 4 h, the hardness decreases slightly, from HV 83 to HV 81; however, it decreases notably from annealing at 250 °C for 4 h to at 300 °C for 4 h, from HV 81 to HV 61, respectively. After annealing at 380 °C for 4 h, the hardness is comparable to that of the as-homogenized sample. This indicates that work hardening can be diminished effectively by annealing heat treatments, which is beneficial for the further development of wide AA 5052 aluminum alloy sheets.



Fig. 8 Variation of hardness with rolling reduction (a) and annealing temperature (b) for AA 5052 aluminum alloy

Figure 9 shows the typical engineering stress-strain curves of AA 5052 aluminum alloy under different conditions at room temperature. Figure 9(a) shows the typical engineering stress-strain curves of AA 5052 aluminum alloys with different rolling reductions. As seen in Fig. 9(a), cold rolling leads to a notable change in mechanical properties of the AA 5052 alloy. For as-cast AA 5052 alloy, the ultimate tensile strength (UTS), yield strength (YS) and elongation are 199 MPa, 69 MPa and 27%, respectively. Both the strength and elongation change a little after homogenized treatment, UTS, YS and elongation are 191 MPa, 70 MPa and 25%, respectively. With increasing rolling reduction, the UTS and YS increase gradually, while the elongation reduces greatly. For the first rolling pass (15% rolling reduction), the elongation is as low as 7.5%. UTS increases from 199 to 325 MPa, and YS increases from 69 to 320 MPa with 87% rolling reduction; however, the elongation is only 2.5% compared with the as-cast 5052 alloy. The trend of strength is quite well consistent with the hardness. Obviously, the improvement of the mechanical strength can be mainly ascribed to the following reasons. On one hand, with increasing rolling reduction, grains are severely elongated along the rolling direction. It is beneficial for forming fiber texture along the rolling direction, which gives rise to the improved tensile strength. And also with increasing rolling reduction, much more secondary phase precipitates play an important role in improving strength by precipitation



Fig. 9 Engineering stress-strain curves of AA 5052 aluminum alloys under different conditions at room temperature: (a) Ascast, homogenized and rolled samples; (b) As-rolled AA 5052 aluminum alloy annealed at various temperatures for 4 h

strength mechanism. On the other hand, with increasing rolling reduction, the work hardening is obvious. This can lead to the increase of dislocations density and substructure, which results in the enhanced tensile strength and reduced elongation.

Figure 9(b) shows the typical engineering stressstrain curves of 75% rolling reduction AA 5052 aluminum alloys annealed at different temperatures for 4 h. As shown in Fig. 9(b), the strength declines while the elongation increases after annealing. And with increasing annealing temperature, the strength decreases but the elongation increases. The strength decreases slightly annealing at 220 to 250 °C, while decreases obviously between 250 and 300 °C. From Ref. [37], the tensile strength decreases slightly and ductility increases a little during the recovery, but tensile strength decreases obviously and ductility increases notably during recrystallization. As seen from Fig. 9(b), the UTS decreases from 277 to 267 MPa and ductility increases from 7.2% to 8.5%, annealing at 220 and 250 °C for 4 h, respectively. While UTS decreases obviously from 267 to 212 MPa and ductility increases from 8.5% to 23.4%, annealing at 250 °C and 300 °C for 4 h, separately. After annealing at 350 °C for 4 h, the elongation of the sample is enhanced from 2.8% to 25.2%. But the elongation changes a little when the annealing temperature is above 350 °C. At the same time, UTS decreases from 311 to 212 MPa, and YS reduces from 201 to 75 MPa. This also indicates that the recrystallization occurs at 300 °C. It is in good consistency with Fig. 8(b). Obviously, the following annealing treatment is beneficial for further development of wide AA 5052 aluminum alloy sheets. Combined with Figs. 9(a) with (b), it is observed that the mechanical properties recover to the values of the as-homogenized sample when the 75% rolled-sample is subjected to the annealing at 300-380 °C for 4 h. It is good correspondence with the variation tendency of hardness in Fig. 8. The reasons for the decreased tensile strength and the enhanced elongation are as follows. Firstly, increasing annealing temperature can give rise to increase of grain size. According to the Hall-Petch relation $\sigma_v \propto d^{-1/2}$, grain size d plays an important role in improving the tensile strength. The smaller the grain size is, the better the mechanical properties are. The refined grains prevent the motion of dislocations from increasing the strength, and the slip systems can be changed by the blocked dislocations. As shown in Fig. 5, with increasing annealing temperature, grain size increases, too. When annealing at 380 °C for 4 h, the grain size is as large as 516 µm. Therefore, the tensile strength is the lowest. Secondly, with increasing annealing temperature, the secondary phase is coarser, which is bad for improving strength. Finally, the recrystallization occurs at 300 °C

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and the density of dislocations decreases, leading to the corresponding change of mechanical properties [12–17,38].

4 Conclusions

1) The grains are elongated along the rolling direction after cold rolling. The main phases of as-rolled AA 5052 aluminum alloy comprise α (Al), Mg₂Si and Al₃Fe. The tensile strength increases remarkably and the elongation decreases evidently owing to the work hardening during the process of cold rolling. When the rolling reduction is up to 87%, the ultimate tensile strength reaches as high as 325 MPa.

2) Annealing treatment results in the precipitation of Mg_2Al_3 phase from the matrix, thus the as-annealed alloy comprises α (Al), Mg_2Si , Al_3Fe and Mg_2Al_3 . There are abundant precipitates existing in AA 5052 aluminum alloys annealed at 250–380 °C, especially at 250 °C. The tensile strength is decreased while the elongation is improved gradually with increasing annealing temperature. The tensile strength and elongation are comparable to those of the as-homogenized alloy as the 75% rolled alloy is subjected to the annealing at 300–380 °C for 4 h.

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冷轧及热处理对 AA 5052 铝合金组织及力学性能的影响

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摘 要:通过光学显微镜、扫描电镜、X 射线衍射分析、硬度实验和拉伸实验等方法,研究不同轧制变形量及后续退火处理对均匀化态 5052 铝合金组织与性能的影响。研究结果表明,随着轧制变形量的增加,等轴晶沿着轧制方向明显地被拉长。由于轧制变形量的增加,加工硬化效应导致合金强度升高,硬度下降。当轧制变形量为 87%时,抗拉强度可达 325 MPa,但是伸长率只有 2.5%。经退后处理后,大量的第二相析出。随着退火温度的升高,第二相析出增多,并且明显弱化加工硬化效应。当经过 300 °C 处理 4 h 后,伸长率可达~23%,抗拉强度降至 212 MPa,此时综合力学性能恢复到均匀化状态。

关键词: AA 5052 铝合金; 冷轧; 退火; 显微组织; 力学性能

(Edited by Xiang-qun LI)