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复合微合金化对 Al–Mg 合金组织与性能的影响 *

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摘 要 研究了 Sc 和 Ti 复合微合金化对 Al–Mg 合金显微组织与拉伸性能的影响. 结果表明: Sc 和 Ti 复合微合金化可以显著提高 Al–Mg 合金的强度, 并可细化铸态合金的晶粒组织. 微量 Sc 和 Ti 的加入可使合金中形成大量细小弥散的球形 Al_3(Ti, Sc) 粒子, 这些 Al_3(Ti, Sc) 粒子对位错和亚晶界具有强烈地钉扎作用, 因而能强烈抑制合金的再结晶. Sc 和 Ti 复合微合金化的 Al–Mg 合金的强化作用主要来源于 Al_3(Ti, Sc) 粒子的析出强化和亚结构强化以及细晶强化.

关键词 金属材料, Al–Mg–Ti–Sc 合金, Al_3(Ti, Sc) 粒子, 显微组织, 力学性能, 微合金化

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Effects of micro–alloying with Sc and Ti on the microstructure and mechanical property of Al–Mg based alloys

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ABSTRACT The effects of micro–alloying with small amounts of Sc and Ti on the microstructure and mechanical property of the Al–Mg based alloys were investigated. It was found that the micro–alloying could significantly enhance the tensile strength of the alloys, and refine their grain size of the cast structure. Large fraction of fine, spherical and dispersive Al_3(Ti, Sc) particles were formed in the annealed Al–Mg–Ti–Sc alloys, which could strongly pin up dislocations and subgrain boundaries, thus strongly retarding the recrystallization of the alloys. The strengthening of the micro–alloyed Al–Mg alloys was attributed to the precipitation strengthening by the Al_3(Ti, Sc) particles, the substructure strengthening and the grain refining strengthening.

KEY WORDS metallic materials, Al–Mg–Ti–Sc alloy, Al_3(Ti, Sc) particle, microstructure, mechanical properties, micro–alloying

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Aluminum alloys containing scandium have a number of excellent properties, such as high strength together with high ductility, good neutron–irradiation and corrosion–resistance, and superior weldability[1~8], thus can serve as a high–performance structural material. However, a critical Sc content in the alloys must be reached in order to obtain a good performance of them[9~11], suggesting that a relatively large quantity of Sc must be added into the alloys. Due to the high price of Sc, and hence to the high cost of the Sc–containing aluminum alloys, the research, development and application of these alloys have been severely retarded. Thus, reducing the Sc addition while keeping the properties of the alloys unchanged has become a key point to the development of the Sc–containing aluminum alloys. Micro–alloying with elements in addition to Sc has been considered as an effective way of both enhancing the properties and reducing the cost of the alloys. In the present paper, the effects of micro–alloying with Sc and Ti on the microstructures and mechanical properties of Al–Mg based alloys are reported, and the mechanism by which the micro–alloying affects the properties of the alloys is elucidated.

1 Materials and experimental procedures

Four alloys for the study (marked A, B, C, D) were prepared by ingot metallurgy, using pure Al, pure Mg and Al–Sc, Al–Ti master alloys , as the starting materials. Their nominal compositions are listed in Table 1. After homogenization at 460 ℃ for 13 h, the ingots were hot–rolled (at a deformation rate of 80%) and then cold–rolled (at a deformation rate of 45%) to 2.5 mm thick plates. The specimen pieces were annealed at 340 ℃ for 1 h or 130 ℃ for 4 h. Tensile specimens were cut along the rolling direction of the plates and tested on a CSS–44100 tensile testing machine.

The microstructures of the alloys were examined using a Polyver–Met optical microscope, with the specimens electro–polished first, followed by anodizing in a water solution of HF and H_3BO_3. TEM thin foils were prepared by twin–jet polishing with an electrolyte solution composed of 30% nitric acid and 70% methanol at a temperature below –20 ℃. The foils were examined using a Hitachi–800 transmission electron microscope at an accelerating voltage of 200 kV.

Table 1 Chemical composition of the 4 alloys studied

Specimen	Chemical composition(mass fraction, %)			
No.	Al	Mg	Sc	Ti
A	Bal.	5		
B	Bal.	5	0.2	
C	Bal.	5		0.04
D	Bal.	5	0.2	0.04

2 Results

2.1 Effect of Sc *and* Ti *on the tensile properties of* Al–Mg *based alloys*

Table 2 lists the tensile properties of the 4 alloys annealed at 130 ℃ for 3 h or at 340 ℃ for 1 h. It is clear that co–addition of small amounts of Sc and Ti into Al–Mg alloys can appreciably improve the tensile strength (σ_b) and yield strength ($\sigma_{0.2}$) of the alloys subjected to the 2 annealing treatments, while still keeping their ductility (δ) at a high level (>13.4%). Specifically, σ_b increases by 44~46 MPa and $\sigma_{0.2}$ by 35~70 MPa, as compared to the Al–Mg alloys; and σ_b increases by 22~27 MPa and 16~41 MPa respectively, $\sigma_{0.2}$ increases by 10~42 MPa and 11~63 MPa, respectively, as compared to the Al–Mg–Sc and Al–Mg–Ti alloys. It is also clear from Table 2 that a better combination of strength with ductility was obtained when annealed at 130 ℃ for 3 h than that at 340 ℃ for 1 h.

Table 2 Tensile properties of the 4 annealed alloys

Heat	σ_b/MPa				$\sigma_{0.2}$/MPa				δ/%			
treatment	A	B	C	D	A	B	C	D	A	B	C	D
130 ℃/3 h	329	348	359	375	240	265	264	275	19.3	15.4	13.6	13.4
340 ℃/1 h	282	304	285	326	124	152	131	194	25.4	21.0	19.4	13.7

2.2 Optical microstructures of the alloys

Fig.1 illustrates the microstructures of the 4 as–cast alloys. The grain size of alloy A is about 370 μm, and those of alloy B and alloy C have decreased to about 308 μm and 184 μm respectively,

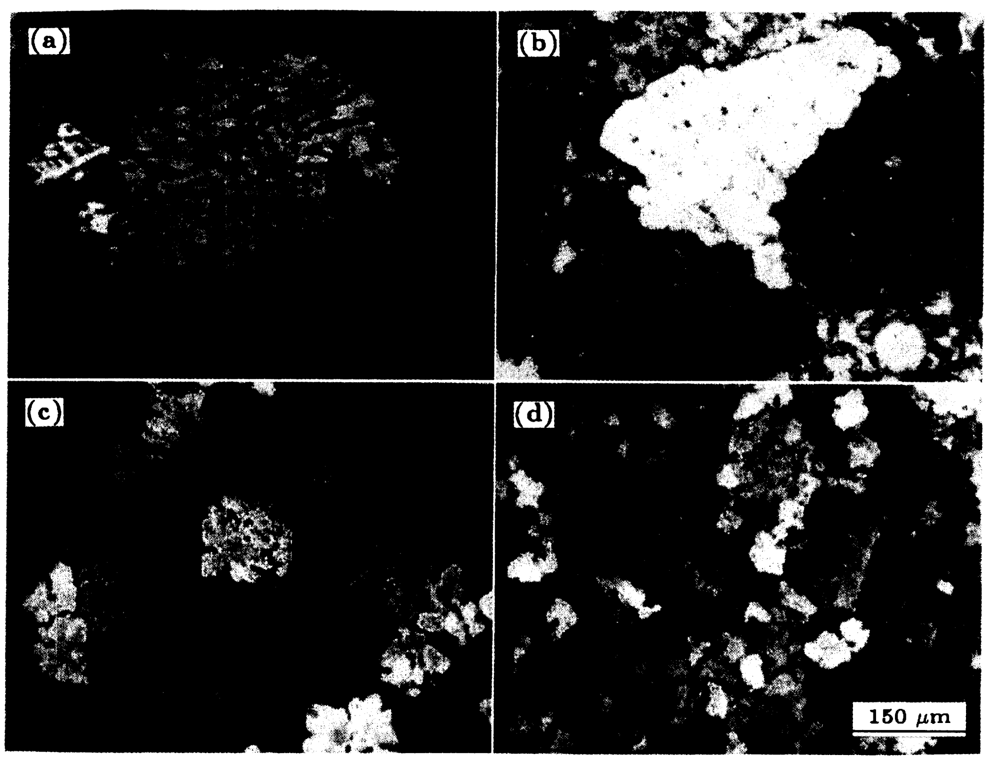

Fig.1 Optical microstructures of the 4 alloys (as–cast)
(a) alloy A, (b) alloy B, (c) alloy C, (d) alloy D

but that of alloy D is only 67 μm. So the gain size of alloy D (Fig.1d) is about 1/6 that of the alloy A(Fig.1a), 1/5 that of the alloy B (Fig.1b), and 1/3 that of the alloy C (Fig.1c), respectively. These observations indicated that addition of 0.04% Ti into Al–Mg based alloys can bring about a grain refining effect of the cast alloys, but co–addition of 0.2% Sc and 0.04% Ti can bring about better effect.

Microstructures of the hot rolled alloys are shown in Fig.2. After hot–rolling at 450 ℃, the Al–5Mg (alloy A) was completely recrystallized, producing fine, nearly equiaxed microstructure (Fig.2a) which however were not observed in the other three alloys. Instead, these alloys possessed a fibrous structure along the rolling direction (Fig.2b, c, d). Sc and Ti co–addition results in finest fibrous structure in alloy D, thus suggesting that the addition of Ti or Sc could retard recrystallization of the Al–Mg alloys during hot–rolling, and the retarding effect was the best when Sc and Ti were co–added.

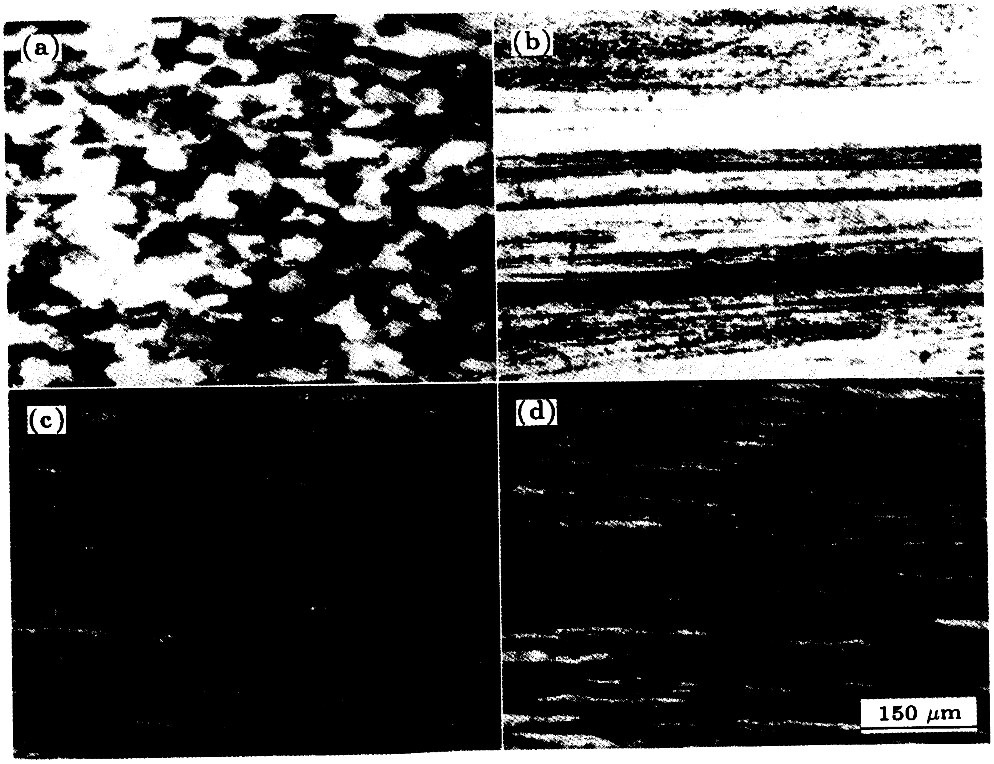

Fig.2 Optical microstructures of four studied alloys (hot–rolled, 80%)
(a) alloy A, (b) alloy B, (c) alloy C, (d) alloy D

The microstructures of the alloys annealed are shown in Fig.3. After annealing at 340 ℃ for 1 h, the Sc–free alloys A and C have been completely recrystallized (Fig.3a, c), with a marked grain growth occurring in alloy A. Alloy B however still keeps the fibrous structure, with a small quantity of fine, nearly equiaxed grains formed in it(Fig.3b), thus producing a partially recrystallized structure. No such recrystallization occurred in alloy D in which the fibrous structure remained unchanged. It is concluded from these observations that the addition of Sc can retard recrystallization during annealing, and the retarding effect is the strongest when Sc and Ti are co–added.

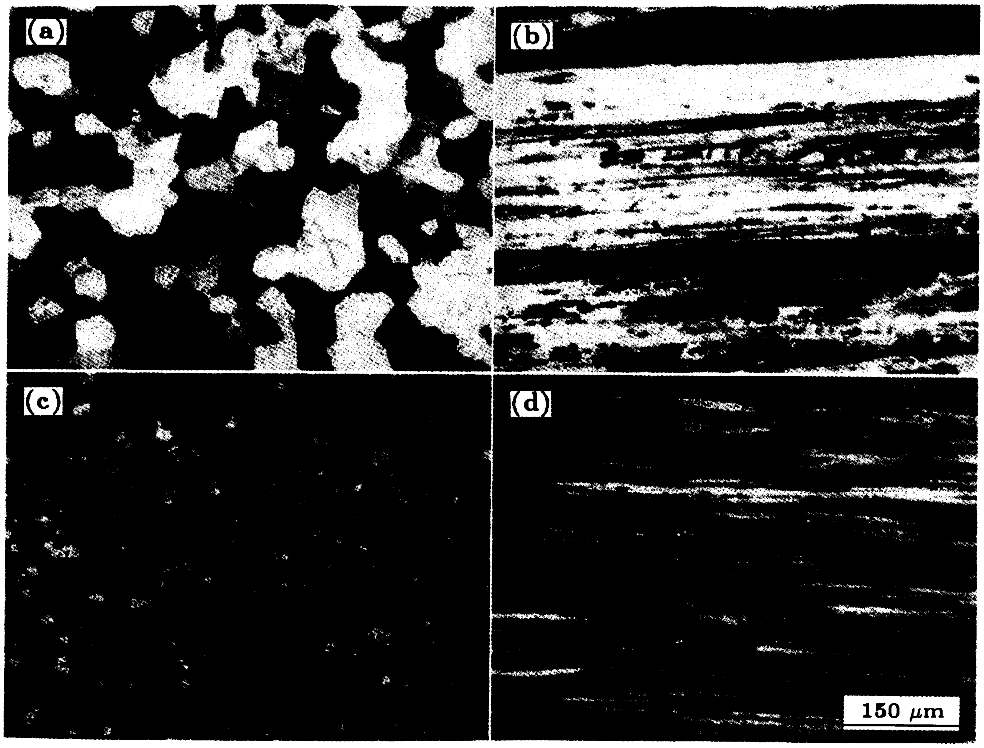

Fig.3 Optical microstructures of the 4 alloys (340 ℃/1 h annealed)
(a) alloy A, (b) alloy B, (c) alloy C, (d) alloy D

2.3 TEM *microstructures of the alloys*

Fig.4 shows the TEM microstructures of alloys D, which annealed at 340 ℃ for 1 h. It can be seen that a large fraction of fine and dispersive particles have precipitated within grains in alloy D (Fig.4a, c), which tightly pin up the dislocations (Fig.4b) and the subgrain boundaries (Fig.4c). These particles are the secondary Al_3(Ti, Sc) precipitates formed during the cooling course after annealing[12~14]. A lot of subgrains are in the alloy which can lead to the sub–structure strengthening (Fig.4c, d).

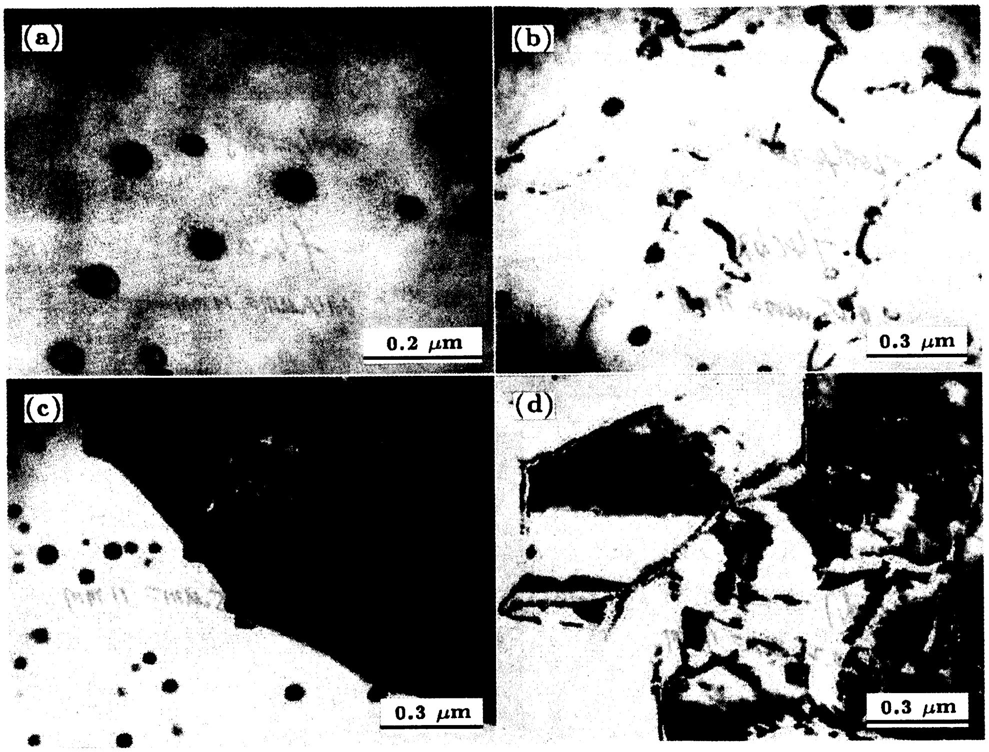

Fig.4 TEM micrographs of alloys annealed at 340 ℃ for 1 h (alloy D)
(a) the fine, spherical and dispersive secondary $Al_3(Ti, Sc)$ particles in alloy D;
(b) the secondary $Al_3(Ti, Sc)$ particles pinning up dislocations;
(c) the secondary $Al_3(Ti, Sc)$ particles pinning up subgrain boundaries in alloy D;
(d) the subgrains in alloy D

3 Discussions

When simultaneously adding minor Sc and Ti were into Al–Mg alloys, a little quantity of Sc and Ti is solution in α Al, and the majority exist in two forms of $Al_3(Ti, Sc)$ intermetallic compound. One is primary $Al_3(Ti, Sc)$ precipitated from the melt during solidification and the other is secondary $Al_3(Ti, Sc)$ precipitated from heat process. The $Al_3(Ti, Sc)$ phase is $L1_2$ structure and its lattice constant is about 0.40701 nm[13]. These are similar to those of the base. And its misfit is little with the base, about 0.5%~1%. So it is coherent with the base Al. Its melting point is about 1559 ℃ [13] and far higher than that of the base(660 ℃). So primary $Al_3(Ti, Sc)$ is an ideal heterogeneous crystal nucleus[14,15], and they can be prior precipitate during solidifying and significantly refine the grain size of Al–Mg as–cast alloys(Fig.1d). This leads to the grain refining strengthening. The secondary $Al_3(Ti, Sc)$ is fine, dispersive and homogeneous (Fig.4a, c). These dispersoids can tightly pin up the dislocations (Fig.4b) and retard their movement during deformation, thus making dislocation sliding more difficult and the stress required for deformation increased. Meanwhile, they can hold up the migration and annexation of subgrain boundaries(Fig.4c and d), thus stabilizing the deformation structures. So they can lead to the precipitation strengthening and subgrain strengthening. The results also show that micro–alloying with Sc and Ti can retard the recrystallization during the hot–rolling and annealing process of the Al–Mg alloy, which also mainly results from the Al_3(Ti, Sc) particles pinning up the dislocations and subgrain boundaries.

The experiment results have showed that micro–alloying with small amounts of Sc and Ti can improve the properties of Al–Mg alloys. Meanwhile the resource of Sc can be saved and then the cost reduced, the high–strength and high–ductility Al–Mg alloys containing Sc can be obtained.

4 Conclusions

1. Micro–alloying Al–Mg alloys with small amounts of Sc and Ti can considerably increase the strength of the alloys, while still keeping their ductility at a high level. The strengthening mainly results from sub–structure strengthening, precipitation strengthening and grain refining strengthening.

2. Co–addition of small amounts of Sc and Ti into Al–Mg alloys can significantly refine the as–cast grain size and retard the recrystallization of Al–Mg alloys during hot–rolling and annealing process, thus stabilizing their deformation structures.

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