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ARTICLE

Effect of Trace Elements La, Ti and Homogenization on Electrical Properties of Pure Aluminum

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Abstract: Al-La, Al-Ti and Al-La-Ti alloys were prepared to study the effects of Ti and La on the electrical conductivity and strength of alloys. The electrical conductivity, strength and microstructure evolution of alloys before and after homogenization treatment at 500 °C for 8 h were investigated. The results show that when the content of La does not exceed 0.3 wt%, the electrical conductivity of Al-La can keep a high level as 60.35% IACS due to the precipitation of the compounds containing La, Si and Fe. The strength of Al-Ti alloy is rapidly enhanced with the increase of Ti due to the increasing content of Ti atoms in the α-Al solution. When La and Ti are added simultaneously, the strength of alloys is higher than that of Al-La alloys, and the electrical conductivity is decreased compared with that of Al-La alloys but is higher than that of the Al-Ti alloy, which is mainly attributed to the formation of the new secondary phase $Ti_2Al_{20}La$. Homogenization treatment has little effect on the microstructure of alloys but reduces the number of casting defects, thereby increasing the electrical conductivity of all samples.

Key words: pure aluminum; La; Ti; homogenization treatment; electrical conductivity

In recent years, aluminum wire is widely used as an electrical conductor in the field of electrical engineering because of its low density, high electrical conductivity, high strength and low cost. It is necessary to produce high electrical conductivity and high strength aluminum wires to meet the needs of industrial production[1]. However, conventional methods for strengthening alloys usually decrease the conductivity, such as alloying, precipitation strengthening and strain hardening. All these methods above will increase the scattering of conduction electrons, which deteriorates the conductivity significantly[2]. As a result, effective methods for improving the strength without compromising too much electrical conductivity are needed. Micro-alloying is one of the most effective ways to achieve high electrical conductivity and high strength of electrical conductors. For instance, the element Ti usually forms Al_3Ti in α-Al solution acting as a substrate for heterogeneous nucleation in the melt to achieve a fine equiaxed grain structure with improved mechanical properties. Zeren et al[3] found that an increase in the Ti content from 0.1 wt% to 10 wt% leads to an increase in hardness and coarsening of the TiAlSi particles of near-eutectic Al-Si. Gao et al[4] found that the increase of Ti content promotes the morphological transformation of TiAlSi from flake-like to block-like, and primary Si is replaced by block TiAlSi particles in hypereutectic Al-Si alloy. A large number of researches involving the effect of Ti on Al alloy mainly focused on its strength properties and refinement effect, but the influence of Ti on the electrical conductivity of Al alloy has seldom been reported. China owns abundant rare earth resources, and rare earth elements like La and Ce are commonly applied to conductivematerials [5-7]. According to the study of Li et al[8], rare earth elements play an important role in deoxidizing, dehydrogenizing and refining crystal grains, thereby improving the mechanical and electrical properties of aluminum conductors. They found that the content of Ce between 0.05 wt%~0.16 wt% in the Al rod is beneficial for electrical conductivity and tensile strength, because Ce reduces the solid solubility of impurity elements Fe and Si in α-Al solution. Yuan et al[9] reported that when La addition exceeds 0.2 wt%, the tensile strength of Al-Mg-Si-Zr-La based on AA6201 decreases and the electrical conductivity improves,

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which could be a compromise between strength and electrical conductivity. Tsai et al[10] found that the tensile strength of A356 alloy is not changed with increasing Ce concentration, but 0.6 wt% Ce addition increases the elongation of the alloy. And 1.0 wt% Ce addition well refines the eutectic silicon and causes the best modification efficiency and mechanical properties of A356 alloy. Lu et al[11] found that trace addition of La (0.05 wt%~0.1 wt%) tends to neutralize the mutual poisoning effect between B and Sr by forming LaB_6, and the optimized La/B weight ratio of 2:1 in Al-11Si-1.5Cu-0.3Mg alloy improves the ultimate tensile strength (270 MPa) and elongation (5.8%).

Homogenization is an essential process for as-cast alloys to change the microstructure and form a more homogeneous structure for further processing[12]. Homogenization is often used to reduce the degree of the segregation and enhance the performances of alloys[13-15]. Wu Yuna et al[12] investigated the effect of homogenization temperature on microstructure and conductivity of Al-0.2Mg-0.36Si-0.3Ce alloy. They found that the electrical conductivity of the alloy increases to 57.3% IACS after homogenization treatment at 560 °C for 6 h, which is 2.7% higher than that of the as-cast alloy, and no decrement of the strength occurs. But the process has little impact on the macro-segregation and the grain size. Zhao et al[16] found that both the electrical conductivity and mechanical properties of Al-0.5Mg-0.4Si-0.2Fe are improved after homogenization treatment, which can be attributed to the evolution of α-Al_8Fe_2Si ternary phases.

It is well known that the addition of Ti to Al alloy can greatly promote the mechanical properties, and a proper content of La added to the Al alloy has a positive effect on its electrical conductivity and mechanical properties. But the influence of simultaneous addition of trace amounts of La and Ti on the electrical conductivity of commercially pure aluminum is rarely discussed. In this paper, the combinatorial effects of trace elements La and Ti on the electrical conductivity and mechanical properties of commercially pure aluminum as well as the corresponding evolution of properties and microstructures in the process of homogenization were systematically investigated.

1 Experiment

Al-La, Al-Ti and Al-La-Ti alloys were prepared by melting appropriate commercially pure aluminum (99.7%), Al-10La and Al-10Ti master alloys in a graphite crucible using an electrical furnace (RJL(Z)-20/35) at 750 °C. After melting, the alloy was degassed and refined by adding a refining agent (ZS-AJ2a), and then poured into the iron mold which was preheated to 200~300 °C. Thereafter, the sample was homogenized at 500 °C for 8 h.

The tensile test was carried out by a universal tensile tester (SY-100) according to GB 13239-2006. The sample was cut into 20 mm×15 mm×10 mm by a wire-electrode cutter and polished in a metallographic polishing machine (PG-2). Then the electrical conductivity of the block samples was measured using an eddy current conductivity meter (7501A). Each reported value of electrical conductivity is an average of three measurements. The content of La was verified by inductively coupled plasma mass spectrometry (ICP-MS, VG PQExCell). The content of Ti and other impurity elements like Si and Fe was tested by optical emission spectrometer (OES, ATLANTIS). Metallographic specimens were prepared by standard procedures.

The scanning electron microscopy (SEM, SU8010) equipped with energy dispersive spectrometer (EDS) system was used to characterize morphology and estimate the chemical composition of samples. X-ray diffraction (XRD, Rigaku DMAX2000) was used to analyze the solubility of race elements in α-Al solution. The scanning speed was controlled at 0.03°/s and the 2θ value was from 5° to 90°. The metallographic samples with and without homogenization were etched by 15 wt% NaOH reagent to observe the micro-pinholes through optical microscopy (OM, 4XC-V).

2 Results and Discussion

2.1 Chemical composition analysis

The actual chemical composition of the melt alloys is presented in Table 1. Among these elements, the content of Si, Fe and other impurity elements meets the standard of GB/T 30552-2014. It can be seen that the actual concentrations of La and Ti in Al-La and Al-Ti alloys are very close to the designed value and present an increasing trend. However, the actual contents of La and Ti are relatively lower than the designed values in Al-La-Ti alloys. Maybe the solid solution atoms La and Ti react with each other to form an unevenly precipitated compound, resulting in a relatively low testing concentration in the measurement.

2.2 Effect of trace elements La and Ti on electrical and mechanical properties of as-cast pure Al

The mechanical properties and electrical conductivity of Al-La, Al-Ti and Al-La-Ti as-cast alloys with different contents of La and Ti are presented in Table 2. Generally, changes in electrical conductivity and strength present opposite trends with increasing concentration of trace elements. It is worth noting that a higher content of La and Ti leads to higher tensile strength and yield strength values but lower electrical conductivity, which accords with the result of Ref.[2].

For Al-La alloys, the electrical conductivity slightly changes when the concentration of La is less than 0.3 wt%, but significantly decreases from 60.35% IACS of Al-0.3La to 57.24% IACS of Al-0.45La, which can be attributed to the precipitation of compounds containing La, Si and Fe, and the increasing solute concentration of La in the α-Al solution[9]. For Al-Ti alloys, with the increasing content of Ti, the tensile strength is increased at the great cost of electrical conductivity. According to Ref.[17], using Matthiessen's rule, the increase in resistivity can reach 31 nΩ·m when the addition amount of Ti is up to 1 wt%, and the influence of Ti in the solid solution accounts for the major part.

Fig. 1 shows SEM images and element map-scanning of Al-0.3La and Al-0.2Ti as-cast alloys. The needle-like, rod-like and coarse secondary phases are shown in Fig.1a. According to EDS analysis, these phases are characterized by AlFeLa compounds. The map-scanning shows that La (Fig.1a_1) and Fe (Fig.1a_2) are slightly segregated in the AlFeLa phases, which indicates the effect of La on promoting the precipitation of Fe. Si was not detected due to the extremely low concentration. Fig.1b shows the SEM image of the as-cast Al-0.2Ti alloy. No apparent differences were found in the electron image and no second phase was detected. It can be seen from map-scanning images that Ti (Fig.1b_1) and Fe (Fig.1b_2) are uniformly distributed in the α-Al solution. The addition of the La element has the ability to precipitate impurities from the α-Al solution, which can improve the electrical conductivity of pure aluminum. However, the addition of Ti does not have this ability.

Compared with the Al-La and Al-Ti alloys, the Al-0.3La-xTi (x=0.1, 0.2, 0.3, 0.4) alloy has a lower electrical conductivity than Al-0.3La but higher than Al-Ti alloys, which can associate with both the solid solution and the precipitation of Ti. When Ti and La are added simultaneously, a new precipitate $Ti_2Al_{20}La$ forms, thus consuming Ti dissolved in the α-Al solution, resulting in a lower solute concentration of Ti in the α-Al solution and in consequence an enhanced electrical conductivity[18]. On the other hand, with the increasing concentration of Ti in Al-La-Ti alloys, more La atoms are consumed to form $Ti_2Al_{20}La$ compounds, which reduce the precipitation of impurity solid solution elements Fe and Si. In this case, the favorable effect of La on improving electrical conductivity is weakened. Most importantly, the increasing content of Ti increases the solid solu- bility, resulting in a decrease in electrical conductivity. Additionally, the formation of $Ti_2Al_{20}La$ has an adverse effect on grain refinement because of the reduction of Al_3Ti, which may

Table 1 Chemical composition of the experimental alloys (wt%)

Sample	La	Ti	Fe	Si	Other elements	Al
Al-0.075La	0.09	-	0.161	0.072	< 0.06	Bal.
Al-0.15La	0.17	-	0.154	0.067	< 0.06	Bal.
Al-0.30La	0.36	-	0.166	0.092	< 0.06	Bal.
Al-0.45La	0.50	-	0.149	0.067	< 0.06	Bal.
Al-0.06Ti	-	0.071	0.160	0.078	< 0.06	Bal.
Al-0.1Ti	-	0.103	0.170	0.075	< 0.06	Bal.
Al-0.2Ti	-	0.166	0.162	0.064	< 0.06	Bal.
Al-0.4Ti	-	0.228	0.162	0.074	< 0.06	Bal.
Al-0.3La-0.1Ti	0.099	0.082	0.129	0.065	< 0.06	Bal.
Al-0.3La-0.2Ti	0.092	0.088	0.133	0.073	< 0.06	Bal.
Al-0.3La-0.3Ti	0.131	0.148	0.121	0.072	< 0.06	Bal.
Al-0.3La-0.4Ti	0.179	0.219	0.192	0.127	< 0.06	Bal.

Table 2 Electrical conductivity (σ) and mechanical properties of Al-La, Al-Ti and Al-La-Ti as-cast alloys with different addition of trace elements La and Ti

Material		Tensile strength/MPa	Yield strength/MPa	Elongation/%	σ/IACS/%
Al-La	Al-0.075La	63	61	30.44	60.35
	Al-0.15La	64.5	57	34.83	60.69
	Al-0.30La	66	61.3	24.33	60.35
	Al-0.45La	73.3	67	35.11	57.24
Al-Ti	Al-0.06Ti	23	9	45.33	55.17
	Al-0.1Ti	76	40	51.00	53.8
	Al-0.2Ti	77.7	67.3	38.33	51.21
	Al-0.4Ti	79	79	41.67	50.17
Al-La-Ti	Al-0.3La-0.1Ti	74.7	38	34.3	56.72
	Al-0.3La-0.2Ti	76	32	31.43	56.03
	Al-0.3La-0.3Ti	78	38	31.43	54.83
	Al-0.3La-0.4Ti	80	39	35.85	52.76

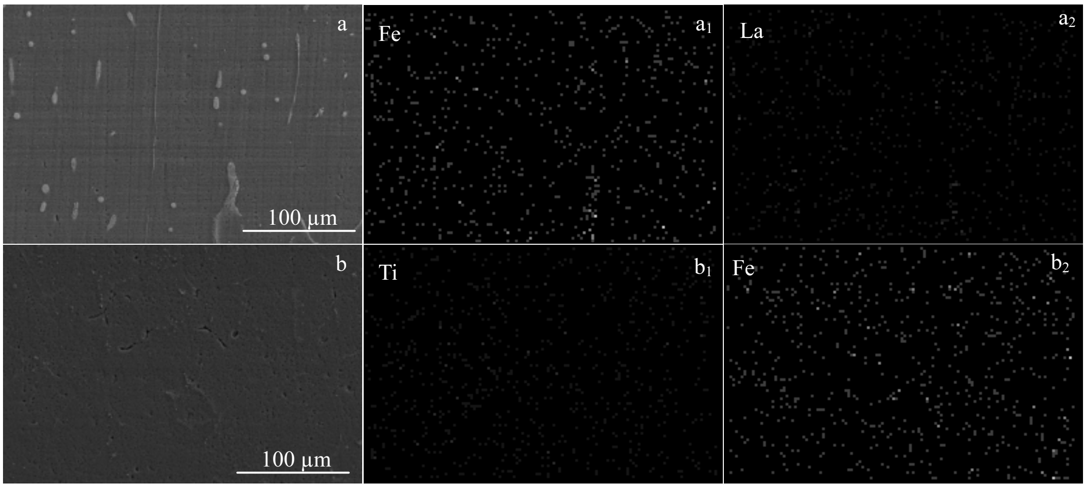

Fig.1 SEM images of Al-0.3La (a) and Al-0.2Ti (b) as-cast alloys and EDS elements map-scanning: (a_1) Fe , (a_2) La of as-cast Al-0.3La and (b_1) Ti , (b_2) Fe of as-cast Al-0.2Ti

partially affect electrical conductivity [18]. It is the combined effect of all these factors that influences the electrical conductivity of alloys.

Fig.2 shows the microstructure features and EDS analysis of as-cast Al-La-Ti alloys with different contents of Ti. Fig.2a shows that with the addition of Ti, the white plate phases precipitate. According to the results of EDS analysis, the second phases are $Ti_2Al_{20}La$. At the same time, a large number of needle-like and strip-like phases precipitate along the grain boundaries and exhibit the network structures, which are characterized by AlFeSiLa phases. Fig.2b shows that when the addition amount of Ti increases to 0.4 wt%, less AlFeSiLa

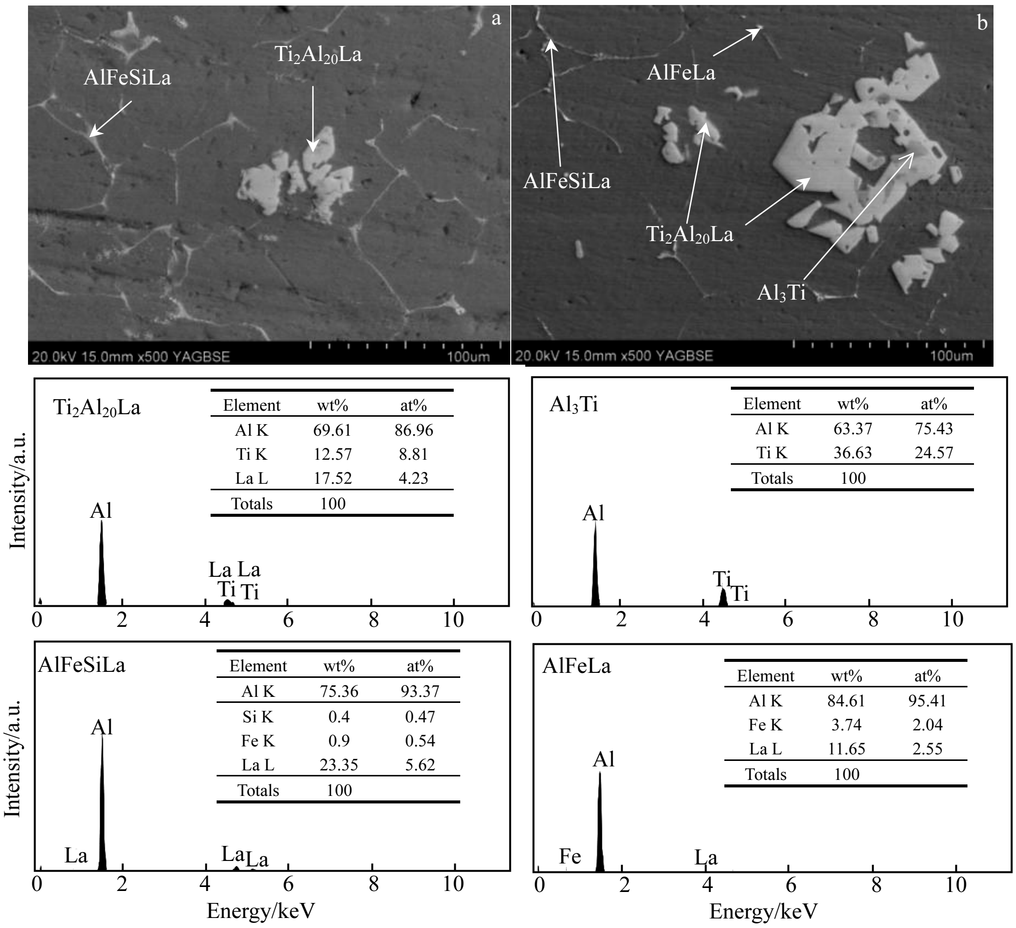

Fig.2 Back scattered electron images and EDS results of as-cast Al-0.3La-0.1Ti (a) and Al-0.3La-0.4Ti (b) alloys

Table 3 Electrical conductivity (σ) and FWHM of the samples before and after homogenization at 500 °C for 8 h

Sample	σ/IACS/%		FWHM/(°)	
	As-cast	Homogenized	As-cast	Homogenized
Al-0.3La	60.35	63.28	0.184	0.182
Al-0.2Ti	51.21	55.29	0.225	0.229
Al-0.3La-0.1Ti	56.72	58.28	-	-
Al-0.3La-0.2Ti	56.03	57.29	-	-
Al-0.3La-0.3Ti	54.83	54.53	-	-
Al-0.3La-0.4Ti	52.76	53.45	0.216	0.211

phases precipitate, and more $Ti_2Al_{20}La$ phase is formed and segregated in the alloy with the gray phases Al_3Ti attaching to the surface of $Ti_2Al_{20}La$. According to Ref.[18-20], the solid solubility of Ti is so low in the α-Al solution that excess Ti reacts with Al to form Al_3Ti which acts as a grain refiner. Meanwhile, when La is added, due to favorable surface absorption energy, La will spontaneously congregate onto the grain boundaries of Al_3Ti to form a core-shell-like $Al_3Ti/Ti_2Al_{20}La$ structure. When Ti and La are added simultaneously, the enrichment of La consumes Ti in Al_3Ti, which causes a low effect of grain refinement.

2.3 Effect of homogenization treatment on electrical properties of the alloys

Al-0.3La, Al-0.2Ti and Al-0.3La-xTi (x=0.1, 0.2, 0.3, 0.4) were chosen as the samples for homogenization treatment. The electrical conductivity of the selected samples before and after homogenization at 500 °C for 8 h is listed in Table 3. The conclusion can be drawn that electrical conductivity of all the homogenized samples is higher than that of the as-cast samples. The existing theory holds that the enhanced electrical conductivity might be attributed to the increasing precipitation of solid solution atoms, and the solid solution atoms have a greater effect on electrical conductivity than precipitation phases in alloys[21,22]. But in the present study, the detailed mechanism was investigated. Thus, XRD and SEM analyses of samples before and after homogenization were performed to analyze the mechanism of enhanced electrical conductivity.

Fig.3 shows XRD patterns of Al-0.3La, Al-0.2Ti and Al-0.3La-0.4Ti before and after homogenization treatment at 500 °C for 8 h. Clearly, no secondary phase is shown in the XRD patterns because of the extremely low concentration of the added elements. However, Ref.[23, 24] figured out that the values of full width at half maximum (FWHM) are reduced to a stable one with the diminution of the lattice distortion caused by solid solution atoms. The FWHM values of the strongest diffraction peak (200) of Al-0.3La and (111) of Al-0.2Ti and Al-0.3La-0.4Ti in XRD with and without homogenization were calculated by Jade and shown in Table 3. It can be noted that almost no change occurs in the FWHM of the homogenized samples compared to the as-cast samples, which proves that there is no obvious change in the solubility of solute atoms in α-Al solution after the homogenization treatment. As a result, the improved electrical conductivity caused by homogenization treatment has little relation with the solubility of the alloying elements.

Fig.4 shows SEM image of the element map-scanning of the homogenized samples. After homogenization treatment, the distribution of Fe, La and Ti is hardly changed compared with that of the as-cast alloys of Al-0.3La and Al-0.2Ti (Fig. 1), but Si is partially segregated in Al-0.3La (Fig. 4a). For Al-0.3La-0.4Ti alloy after homogenization, a large number of second phases containing La and Ti are observed (Fig.4c), with some Al_3Ti phases containing high concentration of Ti elements attached on them. But the microstructure of the homogenized Al-0.3La-0.4Ti alloy has little difference from that of the as-cast sample.

As proved above, the enhanced electrical conductivity of homogenized specimens is seldom caused by the change in solute solubility of alloying elements. There are other reasons in promoting the electrical conductivity. According to Ref.[12], the decreased vacancy concentration is another main contributor,

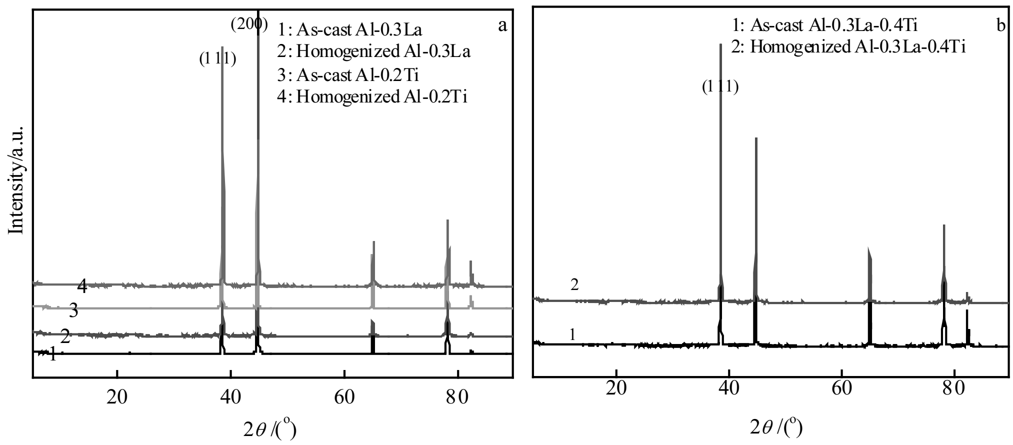

Fig. 3 XRD patterns of Al-0.3La and Al-0.2Ti alloys (a) and Al-0.3La-0.4Ti alloy (b) with and without homogenization treatment

which is directly related to the lattice distortion, thus affecting the electrical conductivity. Therefore, the casting defects like pinholes, porosity and shrinkage of the selected alloys before and after homogenization were observed by optical microscopy and analyzed according to JB/T 7946.3-1999. As shown in Fig. 5a, 5c and 5e, a great number of casting defects are intensively distributed. After homogenization treatment at 500 °C for 8 h, the holes in all specimens are reduced and the size is decreased (Fig. 5b, 5d and 5f), which indicates that homogenization can eliminate the casting defects and in consequence enhance the electrical conductivity. As a result, the reduction in the casting defects like micro-pinholes, micro-porosity and shrinkage by the homogenization treatment is an important reason for the enhanced electrical conductivity.

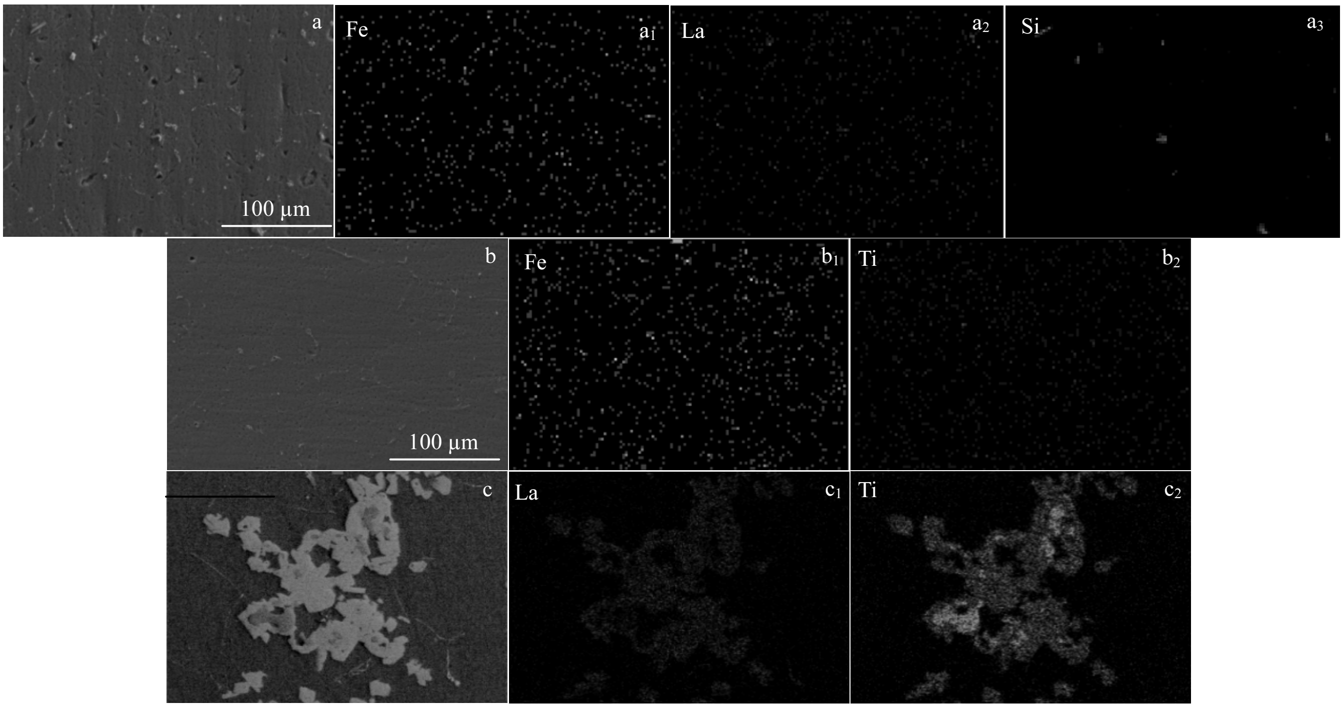

Fig.4 SEM images and the EDS map-scanning of Al-0.3La (a), Al-0.2Ti (b) and Al-0.3La-0.4Ti (c) samples after homogenization at 500 °C for 8 h: (a_1) Fe, (a_2) La, (a_3) Si of Al-0.3La, (b_1) Fe, (b_2) Ti of Al-0.2Ti, and (c_1) La, (c_2) Ti of Al-0.3La-0.4Ti

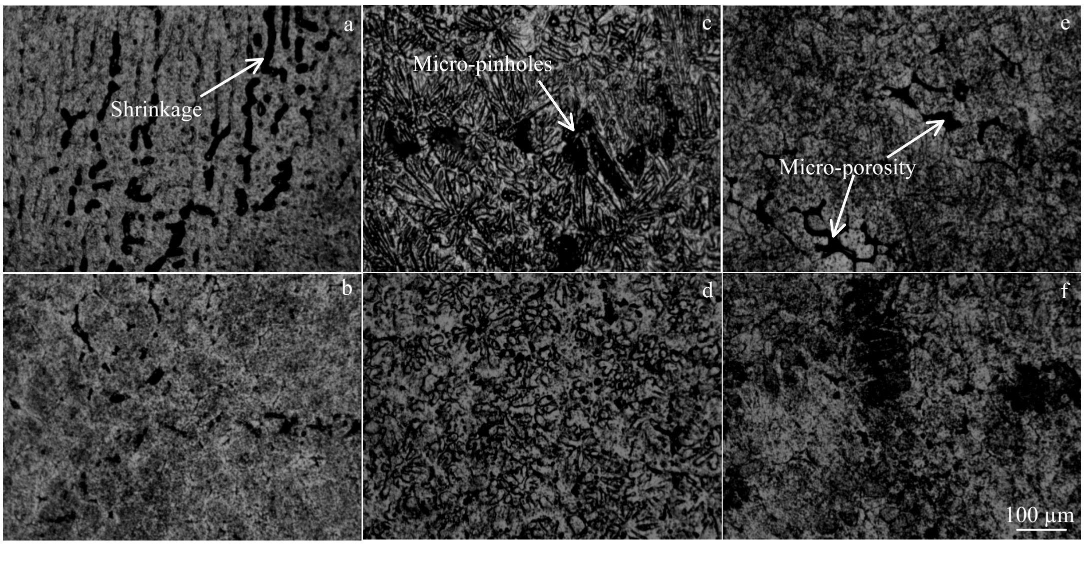

Fig.5 Micro-defects of as-cast Al-0.3La (a), Al-0.2Ti (c), Al-0.3La-0.4Ti (e) alloys and homogenized Al-0.3La (b), Al-0.2Ti (d), Al-0.3La-0.4Ti (f) alloys

3 Conclusions

1) The electrical conductivity of Al-La can keep a high level as 60.35% IACS when the content of La does not exceed 0.3 wt% due to the precipitation of the compounds containing La, Si and Fe. The tensile strength of Al-Ti alloys is rapidly improved and the electrical conductivity is significantly decreased with the increasing addition of Ti. After adding the trace element Ti into Al-0.3La alloy, the tensile strength is enhanced and the electrical conductivity of Al-0.3La-Ti is higher than that of the alloys added with Ti alone, which is mainly associated with the precipitation of new phase of $Ti_2Al_{20}La$. Simultaneously adding trace elements La and Ti into pure Al can achieve a compromise between electrical conductivity and alloy strength.

2) The homogenization treatment is beneficial to the electrical conductivity of Al-0.3La, Al-0.2Ti and Al-0.3La-Ti, which has little relation with the microstructure evolution, but is mainly attributed to the reduced casting defects such as micro-pinholes, micro-porosity and shrinkage in the microstructure after homogenization treatment.

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微量元素 La、Ti 及均匀化处理对纯铝导电性能的影响

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摘 要：通过熔炼制备 Al-La, Al-Ti 及 Al-La-Ti 合金, 研究了 La、Ti 微量元素添加对纯铝的导电性和强度的影响。同时研究了在 500 ℃下均匀化处理 8 h 之后样品的导电性能与微观结构变化。结果表明，当 La 添加量不超过 0.3%(质量分数)时，Al-La 合金的导电率会保持在较高的值 60.35% IACS 左右，因为 La 与铝中的杂质 Si，Fe 反应析出。随着 Ti 元素添加量的增加，Al-Ti 合金的强度明显增加，这主要是 Ti 原子的固溶强化的作用。同时在纯铝中添加 La 和 Ti 元素时，会使合金在 Al-La 合金的基础上强度得到提升，导电率有所下降，但是导电率仍然高于单独添加 Ti 元素时的 Al-Ti 合金。主要是因为 La 与 Ti 之间相互反应生成 $Ti_2Al_{20}La$ 化合物并且析出，有利于导电性的提升。结果表明，均匀化处理对样品微观结构变化的影响很小，而导电性的提高主要是因为均匀化处理消除了铸态样品中大部分显微疏松，缩孔和显微针孔等铸造缺陷。

关键词：纯铝；La；Ti；均匀化处理；导电性

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