Microstructure and mechanical properties of an Al-11La-6Mg alloy prepared by the melt spinning and spark plasma sintering

Xinkui Zhang1,a, Liejun Li1,b*, Zhenwu Peng1,c, Jixiang Gao2,d

1National engineering research center for near-net-shape for metallic materials, south china university of technology, 510640, Guangzhou, P. R. China
2Guangdong Polytechnic Normal University, 510635, Guangzhou, China
aemail: Xinkuizhang1996@163.com
b*Corresponding author email: Liliejun@scut.edu.cn
cemail: pengzw@scut.edu.cn
demail: gaojx@gpnu.edu.cn

Abstract: In order to refine the precipitate and grain, the combination of melt spinning and spark plasma sintering were used to prepare Al-11La-6Mg alloy. The results show that melt spun ribbon consists of some coarse precipitates. After spark plasma sintering, there are dual-scale precipitates in the matrix. As a result, the alloy exhibits a high compressive strength of 933 MPa and strain of 34% at room temperature. Unfortunately, the strength, at 300℃, is inferior to as-cast sample due to the fine grain.

1. Introduction
Aluminum alloys is a common structural material because of its high specific strength and corrosion resistance. However, the development of high temperature aluminum alloys mainly focuses on Al-Si alloys [1].

Due to the excellent high temperature mechanical properties of Al-Re (La, Ce) alloys, they have aroused extensive attention [2]. However, many studies focus on the cast and additive manufacturing Al-Re alloys [3, 4]. Melt spinning is one of the rapid solidification methods, which is cost effective compared with gas atomization and spray forming [5]. And the spark plasma sintering (SPS) is a rapid sintering process, which can effectively resist grain coarsening [6]. However, limited work uses the combination of melt spinning and SPS to fabricate bulk Al-Re alloys. Therefore, in the present work, we successfully prepared high-strength bulk Al-11La-6Mg alloy. The microstructures and mechanical properties at room temperature (RT) and 300℃ are systematically studied.

2. Materials and Methods
Firstly, the cast Al-11La-6Mg alloy were melted at 600℃ by induction heating under a high purity argon atmosphere. And then the melt was ejected on the copper wheel surface with a surface speed of 35 m/s. The process of melt spinning is performed in the machine shown in fig. 1. The ribbons were cut into flakes and then filled into a metal die with a diameter of 30 mm. Cylinder-shape billets were produced by isostatic cold pressing for 1 min. Finally, the billets were sintered by SPS (Dr. Sinter Model SPS-825 Spark Plasma Sintering System, Sumitomo Coal Mining Co. Ltd., Japan). The sintering temperature, holding time and sintering pressure is 475℃, 10 min and 30 MPa respectively.
X-ray diffraction (XRD) was carried out to analyze the phase composition. And the microstructures were observed by scanning electron microscopy (Zeiss Sigma 300) with an energy dispersive spectrometer (EDS).

Compressive testing on cylindrical specimens, with a diameter of 3 mm and high of 6 mm, was conducted on MTS C45.105 at room temperature and 300℃. For compressive tests at 300℃, the specimens were preheated to 300℃ and held for 15 min. The strain rate of RT and 300 ℃ compressive test were set as 1x10⁻³ s⁻¹.

3. Results & Discussion

3.1 Microstructure characterization

Fig. 2. shows the XRD patterns of melt spun ribbons and bulk Al-11La-6Mg alloy. It can be observed that the phase composition of the strip and bulk Al-11La-6Mg alloy are mainly α-Al and Al11La3 phases. Specifically, the peak intensity of the Al11La3 phase in the melt spun ribbon is relatively small, while the peak intensity of the Al11La3 phase in the bulk sample is relatively strong. Similarly, the α-Al peak intensity is also stronger for bulk samples and weaker for strips. This may be due to the fact that the amount of the ribbons is smaller than the bulk Al-11La-6Mg alloy during measurement, resulting in the overall peak diffraction intensity of the bulk Al-11La-6Mg alloy being larger than that of the ribbons.

SEM was employed to investigate the morphologies of melt spun ribbon and bulk Al-11La-6Mg alloy under backscatter electron (BSE) mode. Fig. 3. show the morphologies of ribbon and bulk sample. It can be seen that the bright white precipitates are the Al11La3 phase, and the gray matrix is the α-Al phases. Most of the coarse intermetallic phase in the ingot can be refined because of the high cooling rate during the process of melt-spinning. However, it can be seen from Fig. 3. that there are still a few of coarse precipitates that have not been refined. The reason is that the preheating temperature during the melt spinning process is not high enough, which induces that the large second phase is not completely dissolved. Hence, a few of large precipitates still appear after the rapid solidification process.
Fig. 2. XRD patterns of melt-spun ribbons and SPS bulk sample.

Fig. 4 demonstrates the SEM images of the bulk Al-11La-6Mg alloy under back scattering electron mode. It can be observed that the precipitates in the sample exists in a dual-scale form. Fig. 4b and c are partial enlarged views of the fine and coarse second phase respectively, in which the fine second phases are uniformly distribute on the α-matrix, and some of the coarsen second phases distribute in the form of band-like distribution in the α-Al matrix. The dual-scale form is attribute to the low preheating temperature during the melt-spinning process. The EDS mapping of the fine precipitate region is shown in Fig. 5, where it can be observed that the elements are distributed uniformly.

Fig. 3. SEM image of melt-spun ribbon under BSE mode
3.2 Mechanical properties

Fig. 6 shows the room temperature and high temperature compressive curves of the as-cast sample and SPS sintered samples. The bulk Al-11La-6Mg alloy, prepared with melt spinning and SPS sintering, have great performance at RT, whose compressive strength and strain are 933 MPa and 34%, respectively. In comparison with as-cast sample, the compressive strength and strain have increased by 306 MPa and 7% respectively because of the high cooling rate of melt spinning and the rapid sintering which can induce grain refinement. Hence, not only the compressive strength is greatly improved but also the strain. However, it is that the grains of SPS are greatly refined after sintering, which leads to its poor compression performance at 300℃. At elevated temperature, the grain boundary is weaker than that of the intragranular. Hence, during compression, the bulk samples will yield early at 300℃.

4. Conclusions

This work provides a novel method to fabricate high-strength Al-11La-6Mg alloy. Microstructures were
characterized by XRD and SEM. The mechanical properties were measured by compressive test at RT and 300°C. Following conclusion can be obtained:

1) Most of the precipitates are refine after melt spinning but there are some coarse precipitates in the matrix.

2) Spark plasma sintering results in dual-scale precipitates in the matrix. The regions of fine precipitates exhibit homogenous elements distribution.

3) The sintered samples exhibit a compressive strength of 933 MPa and a strain of 33% at RT. However, the high-temperature mechanical properties are inferior to the as-cast sample. Both the mechanical performance at RT and 300°C are attributed to the refinement of grain and precipitates.

Although the room temperature has been improved by melt spinning and spark plasma sintering, the high-temperature strength is low. Hence, in the future, it is necessary to improve the high-temperature strength.

Acknowledgments

The authors gratefully acknowledge the Major Project of Department of Education of GuangDong Province (2017GCZX003).

References

[1] E.R. Wang, X.D. Hui, G.L. Chen (2011) Eutectic Al–Si–Cu–Fe–Mn alloys with enhanced mechanical properties at room and elevated temperature, Materials & Design 32(8-9) 4333-4340

[2] Z.C. Sims, O.R. Rios, D. Weiss, P.E.A. Turchi, A. Perron, J.R.I. Lee, T.T. Li, J.A. Hammons, M. Bagge-Hansen, T.M. Willey, K. An, Y. Chen, A.H. King, S.K. McCall (2017) High performance aluminum–cerium alloys for high-temperature applications, Materials Horizons 4(6) 1070-1078.

[3] A. Plotkowski, K. Sisco, S. Bahl, A. Shyam, Y. Yang, L. Allard, P. Nandwana, A.M. Rossy, R.R. Dehoff (2020) Microstructure and properties of a high temperature Al–Ce–Mn alloy produced by additive manufacturing, Acta Materialia 196 595-608.

[4] Y. Liu, R.A. Michi, D.C. Dunand (2019) Cast near-eutectic Al-12.5 wt.% Ce alloy with high coarsening and creep resistance, Materials Science and Engineering: A 767

[5] L. Katgerman, F. Dom (2004) Rapidly solidified aluminium alloys by melt spinning, Mater. Sci. Eng. A 375e377 1212e1216.

[6] Z.A. Munir, U. Anselmi-Tamburini, M. Ohyanagi (2006) The effect of electric field and pressure on the synthesis and consolidation of materials: a review of the spark plasma sintering method J. Mater. Sci., 3, pp. 763-777