

Semi-solid thixoforming, microstructure, and mechanical properties of Al-6wt%Si alloy via liquid reaction sintering of elemental powders

Hu Lianxi^{1,a,*}, Liu Wenchao^{1,b}, Sun Yu^{1,c} and Ba Meiyi^{1,d}

¹School of Materials Science and Engineering, Harbin Institute of Technology, China

^ahulx@hit.edu.cn

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Abstract. Al-6wt%Si semi-solid alloy billets were prepared via the liquid phase reaction sintering process by using elemental Al and Si powders as starting materials. Cup-shaped components were fabricated by direct semi-solid thixoforming at 685-595°C with the liquid phase fraction in the range of 0.141-0.196. By microstructure observation and mechanical testing, the influence of the liquid phase sintering and subsequent thixoforming conditions on the microstructure evolution and mechanical properties of the final components was investigated. The results show that, when the liquid phase fraction of the alloy billet is in the range of 0.141-0.167, cup-shaped components with uniform microstructure and no forming defects can be obtained by direct thixoforming. Due to the strengthening effect of the fine Al grains and the Si particles and precipitates, the component thixoformed by using billet with a liquid phase fraction of 0.167 presented the best mechanical properties, with yield strength, ultimate tensile strength and elongation achieving 115MPa, 220MPa and 38%, respectively. It is evident that the semi-solid thixoforming process proposed in the present study turns out to be a practical way to fabricate high-performance Al-Si alloy components.

Introduction

Al-Si alloys are conventionally formed by liquid casting or solid-state plastic deformation [1]. However, casting defects such as segregation and porosity can seriously reduce the quality and performance of the formed parts [2]. Meanwhile, geometrically complicated components are difficult to be formed by forging with low energy consumption and simple processes. Semi-solid forming, a process developed by Flemings in the 1970s [3], can avoid the shortcomings in casting and forging processes. It is believed that semi-solid forming is a promising technique for aluminum alloys because it is an effective way to achieve near-net forming and high-performance [4]. Zhao [5] pointed out that the key to semi-solid forming is to prepare semi-solid alloys with equiaxed solid particles uniformly dispersed in the liquid phase. Takagi [6], Liu[7], and Khosravi [8] reported that semi-solid slurries of Al-Si alloys were prepared by using mechanical stirring, magnetic stirring, and cooling slope method, respectively. However, such semi-solid slurries are difficult to handle with and transport, and their microstructure often contains dendrites. To avoid the aforementioned issues, Shabeshari[9], Bolouri[10], and Shayegh[11] tried ways to prepare semi-solid alloys by strain induced melt activation (SIMA), recrystallization and partial re-melting (RAP), respectively. However, these methods require the casting alloy to undergo deformation treatment and isothermal heat treatment in sequence, which increases production costs and makes the production process more complex. Therefore, we have developed a new process, liquid-phase reaction sintering of powder compacts, to prepare semi-solid Al-Si alloys [12-14]. It generates Si-rich Al liquid phase by alloying reaction between Si and Al elemental powder particles during sintering. The Si-rich Al liquid phase resulting from the alloying reaction of Si and Al surrounds the partially melted solid Al particles to form a semi-solid microstructure.



So far, no reports have been seen that Al-Si alloys parts are fabricated by thixoforming of liquid phase sintered semi-solid powder compacts. In the present study, the liquid phase reaction sintering method was used to prepare Al-6wt%Si semi-solid alloy billets by using elemental Al and Si powders as starting materials, and cup-shaped components were fabricated by direct semi-solid thixoforming following liquid phase reaction sintering. The influence of the semi-solid billet preparation and thixoforming conditions on the microstructure evolution and mechanical properties of the final components was investigated.

Experimental

The process of semi-solid thixoforming of Al-6wt%Si alloy cup-shaped components is shown in Fig. 1. It consists of three steps: (1) cold compaction of mixed Al and Si elemental powders to prepare a green compact by using a hydraulic press with maximum nominal load of 600kN; (2) preparation of the semi-solid alloy billet by liquid phase reaction sintering of the green compact by using an electric furnace with argon protection; (3) Fabrication of cup-shaped component by direct semi-solid thixoforming (backward extrusion) of the as-sintered billet. For the convenience to clamp the semi-solid billet and to prevent the liquid phase from leaking during sintering, the thixoforming temperature was chosen to be at 685-595°C with the liquid phase fraction in the range of 0.141-0.196 [15], and under pressure of 100MPa by using a standard hydraulic machine with maximum nominal load of 1000kN.

After thixoforming, microstructure observation and mechanical testing experiments were carried out to investigate the influences of the liquid reaction sintering and the subsequent semi-solid thixoforming parameters on the microstructure evolution and mechanical properties of the final components. The mechanical properties were characterized by room-temperature compression tests (one sample for each liquid phase fraction) and hardness tests (three samples for each liquid phase fraction). The compression and hardness tests were carried out by using an Instron-5500R machine and a HVS-500 Vickers hardness tester, respectively. The microstructure of the thixoformed components was characterized by metallographic microscopy using a Zeiss Imager A2m optical microscope.

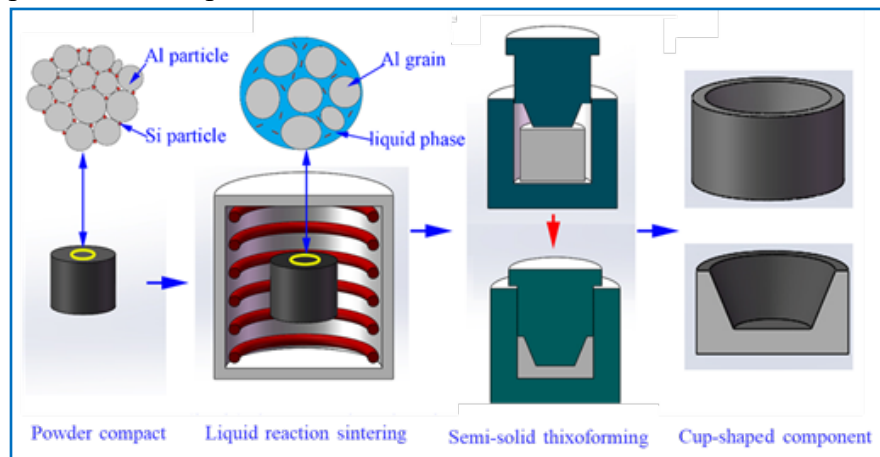


Figure 1 – The process of semi-solid thixoforming of Al-Si alloy cup-shaped component

Results and discussion

Microstructure. Fig. 2 shows the as-sintered Al-6wt.%Si alloy semi-solid billets (Fig. 2(a)) and the subsequently thixoformed components (Fig. 2(b)) with the starting liquid phase fraction during thixoforming in the range of 0.141-0.196, respectively. In all cases, components with uniform thickness and no forming defects such as apparent cracking or porosity were obtained, as is shown by the photos of the components cross-cut open in Fig. 2(b). This indicates that the thixoforming

experiments were very successful and the Al-6wt.%Si semi-solid alloy presented very good deformability when its liquid phase fraction is in the range of 0.141-0.196.

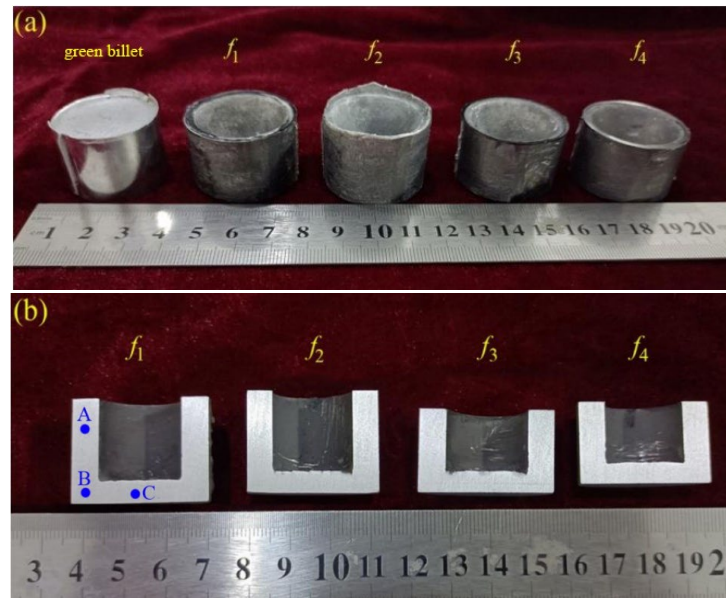


Figure 2 – Al-6wt.%Si semi-solid billets (a) and thixoformed components (b) with different liquid phase fraction: $f_1=0.141$; $f_2=0.156$; $f_3=0.167$; $f_4=0.196$

To further investigate the microstructure of the thixoformed components, microstructural observations were made at positions A, B, and C in components thixoformed with different liquid phase fraction, as shown in Fig. 2 (b). Fig. 3 shows the microstructure at different positions, A, B, and C of the Al-6wt.%Si alloy cup-shaped component thixoformed with a starting liquid phase fraction of 0.141. The microstructure at different positions is featured with typical semi-solid characteristics, that is, α -Al grains (solid phase in the starting semi-solid alloy) are uniformly dispersed and surrounded by the fine Al/Si phases (mostly Al-Si eutectic phases) formed during thixoforming by solidification of the Al-Si liquid phase in the semi-solid alloy. The fact that the α -Al grains at positions A, B, and C are all equiaxed and with very similar average grain size suggests that the solid phase in the semi-solid billet have undergone little or no plastic deformation during the backward extrusion thixoforming. In addition, no microstructural defects such as flow instability, micro-cracking or porosity were found, attributed to the high tri-axial compressive stress state everywhere in the billet during the back-ward extrusion thixoforming process.

Fig. 4 shows the effect of liquid phase fraction (changing from 0.156 to 0.196) of the semi-solid billet on the microstructure at position B of the cup-shaped component by thixoforming. The as-thixoformed microstructures by using billets with different liquid phase fractions are similar in that the α -Al phase grains are equiaxed, indicating that the deformation of the solid phase is in general negligible and the semi-solid alloy deforms mainly by solid-phase grain sliding and liquid-phase flow during the thixoforming process. However, due to the fact that a higher liquid phase fraction is equivalent to a higher thixoforming temperature, which leads to coarser starting solid α -Al grains in the semi-solid billet, the average size of the α -Al grains in the final component therefore increases with increasing billet liquid phase fraction. On the other hand, a higher liquid phase fraction helps to improve the rheological deformability of the semi-solid alloy. Therefore, appropriately increasing the liquid phase fraction in the semi-solid alloy billet during thixoforming

helps to reduce the thixoforming load needed to ensure full die filling, eliminate forming defects, and improve the component quality.

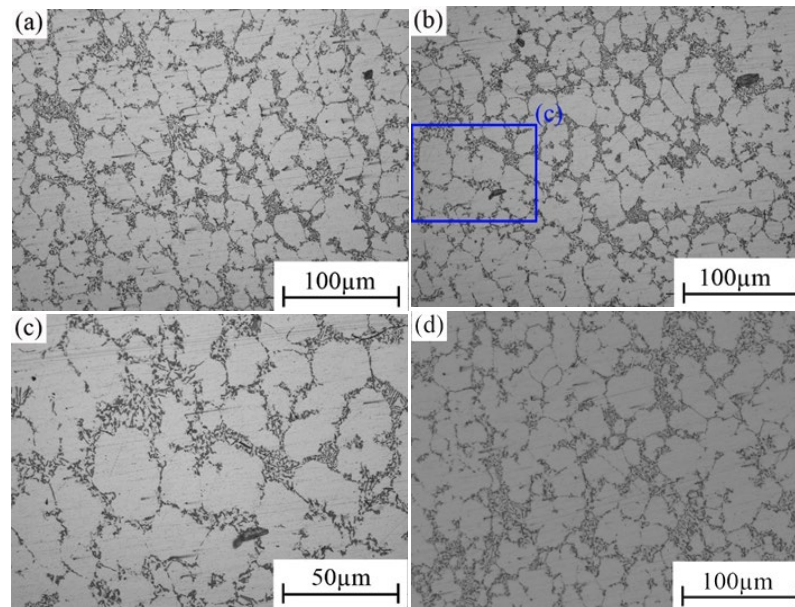


Figure 3 – Microstructure of Al-6wt.%Si alloy cup-shaped component thixoformed with liquid phase fraction $f_l=0.141$: (a) position A, (b) and (c) position B, (d) position C

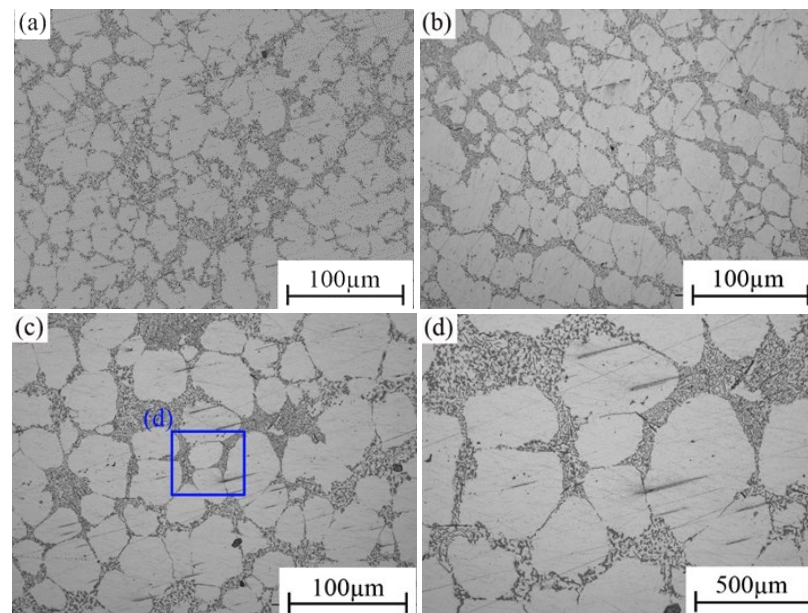


Figure 4 – Microstructure of Al-6wt.%Si alloy cup-shaped components thixoformed with different liquid phase fractions: (a) $f_2=0.156$, (b) $f_3=0.167$, (c) and (d) $f_4=0.196$

Mechanical property. The hardness of the α -Al grains (solid phase in the starting semi-solid alloy) and the fine Al/Si phases (mostly Al-Si eutectic phases) by solidification of the liquid phase during thixoforming in different components was tested separately, and the results are shown in Fig. 5 (a). It is seen that, with the increase of liquid phase fraction in the semi-solid alloy billet, the hardness of the α -Al grains in the component basically keeps steady, with its value being around 35Hv, which is slightly higher than that of pure Al phase, for all tested samples. The hardness of the fine Al/Si phases formed in the components by solidification of the liquid phase during thixoforming is around 80Hv for all samples, being also independent of the liquid phase fraction in the semi-solid alloy billet. That is to say, the hardness of the fine Al/Si phases is much

higher than that of the α -Al grains, but it is not affected by the liquid phase fraction in the semi-solid alloy billets.

According to the characteristics of the liquid phase reaction sintering process, it is sure that, when the compact with mixed Al and Si elemental powders is heated to the temperature above the solidus of the alloy, solid Al particles react partially with fine Si particles to form the Si-rich liquid Al phase, and the retained solid Al particles form α -Al solid phase grains in the semi-solid Al-6wt%Si alloy. The liquid phase contains a large amount of Si element, and it will transform into fine α -Al(Si) solid solution phases and Al-Si eutectic phases by solidification during thixoforming, leading to solid solution strengthening and Orowan strengthening. Therefore, the hardness of the fine Al/Si phases formed by solidification of liquid phase during thixoforming present much higher strength and hardness than that of pure Al matrix [13].

The compressive mechanical properties of the thixoformed Al-6wt%Si alloy components were tested by compression tests at room temperature. The compression samples were cut from the side wall of the cup-shaped components, with dimensions of $\Phi 4\text{mm}$ in diameter and 6mm in height. The results of compression tests are shown in Fig. 5 (b). It is seen that the strength is mainly related to the billet liquid phase fraction and the starting α -Al grain size. Due to the effect of solid solution strengthening and Orowan strengthening, the Al/Si phases formed by solidification of the liquid phase during thixoforming present much higher strength than the starting solid α -Al grains, and increasing the liquid phase fraction in the semi-solid alloy billet will lead to an increase in alloy strength. On the other hand, refining of the starting solid α -Al grains also helps to increase the alloy strength due to Hall-Petch relationship. It seems that the component thixoformed with a liquid phase fraction of 0.167 ($f_3=0.167$) has the best combination of starting α -Al grain size and the amount of fine Al/Si phases by solidification of liquid phase during thixoforming, and thus possesses the best comprehensive mechanical properties, with the yield stress, the ultimate compressive strength, and compressive failure strain achieving 115MPa, 220MPa, and 0.45, respectively. The thixoforming experiments show that, when the liquid fraction in the semi-solid billet is around 0.167, the cup-shaped component has the best mechanical properties. That is to say, the optimized liquid phase fraction for thixoforming of Al-6wt%Si semi-solid alloy prepared by liquid phase sintering of Al/Si powder compact is 0.167.

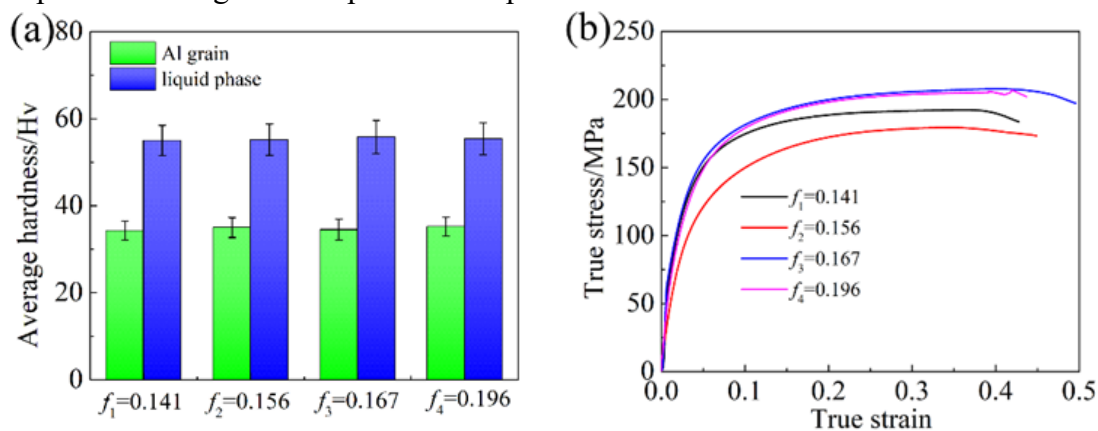


Figure 5 – Mechanical properties of Al-6wt%Si alloy cup-shaped components by thixoforming: (a) hardness, (b) compressive stress-strain curves

Conclusions

(1) The Al-6wt%Si semi-solid alloy billets were prepared by liquid-phase reaction sintering using Al and Si elemental powders as starting materials. Cup-shaped components with high performance and no defects were fabricated by direct thixoforming of the as-sintered semi-solid alloy billets with liquid phase fraction in the range of 0.141-0.196.

(2) Both the size of the solid α -Al grains and the liquid phase fraction in the Al-6wt%Si semi-solid alloy billets have significant impact on the mechanical properties of thixoformed cup-shaped components. Increasing the liquid phase fraction or refining the solid α -Al grains in the semi-solid alloy billet helps to increase the mechanical strength of the thixoformed components. On the other hand, promoting the liquid phase fraction also leads to coarsening of the solid α -Al grains. Therefore, the cup-shaped component thixoformed by using billet with a liquid phase fraction of 0.167 presented the best mechanical properties, with its yield stress, ultimate compressive strength, and compressive failure strain achieving 115MPa, 220MPa, and 0.45, respectively, attributed to the optimal balance between the size of the α -Al grains and the amount of fine Al/Si phases formed by solidification of the liquid phase.

(3) The present study demonstrates that liquid phase reaction sintering of powder compact with mixed elemental powders as starting material is a practicable way to prepare high-quality semi-solid Al-Si alloy billet. The combination of liquid phase sintering and thixoforming, as a new technique, shows good prospect in manufacturing of high-performance complicated Al-Si alloy components.

References

- [1] W Qu, D Li, F Zhang. Multiphase modelling of the transient flow for Sn-15Pb and 3570 alloys in semi-solid die casting process, J Mater Process Tech. 278 (2020) 116534. <https://doi.org/10.1016/j.jmatprotec.2019.116534>
- [2] Y G Wang, Q T Guo, N Ren, W Zhang. Experimental and simulation studies of layer casting on industrial-scale Al-4.5wt Pct Cu ingot: Effect, defect, and optimization, J Mater Process Tech. 32 (2003) 118205. <https://doi.org/10.1016/j.jmatprotec.2023.118205>
- [3] Flemings M C. Behavior of metal alloys in the semisolid state, Metallurgical Transactions B. 22 (1991) 269-93. <https://doi.org/10.1007/BF02651227>
- [4] M Kiuchi, R Kopp. Mushy/Semi-Solid Metal Forming Technology-Present and Future, CIRP Annals. 51 (2002) 653-70. [https://doi.org/10.1016/S0007-8506\(07\)61705-3](https://doi.org/10.1016/S0007-8506(07)61705-3)
- [5] Z Zhao, Q Chen, Y Wang. Microstructural evolution of an ECAE-formed ZK60-RE magnesium alloy in the semi-solid state, Mat Sci Eng A-Struct. 506 (2009) 8-15. <https://doi.org/10.1016/j.msea.2008.12.042>
- [6] H Takagi, Y Uetani, M Dohi. Effects of Mechanical Stirring and Vibration on the Microstructure of Hypereutectic Al-Si-Cu-Mg Alloy Billets, Materials Transactions. 48 (2007) 960-6. <https://doi.org/10.2320/matertrans.48.960>
- [7] D Liu, H V Atkinson, H Jones. Thermodynamic prediction of thixoformability in alloys based on the Al-Si-Cu and Al-Si-Cu-Mg systems, Acta Materialia. 53 (2005) 3807-19. <https://doi.org/10.1016/j.actamat.2005.04.028>
- [8] H Khosravi, R Eslami-Farsani, M Askari-Paykani. Modeling and optimization of cooling slope process parameters for semi-solid casting of A356 Al alloy, T Nonferr Metal Soc. 24 (2014) 961-968. [https://doi.org/10.1016/S1003-6326\(14\)63149-6](https://doi.org/10.1016/S1003-6326(14)63149-6)
- [9] S G Shabestari, M Abdi, S Naghdali. Effect of thixoforming and precipitation hardening on microstructure and mechanical properties of Al-10.5Si-3Cu-0.2Mg alloy produced by strain induced melt activation process, J Mater Res Technol. 15 (2021) 4981-4992. <https://doi.org/10.1016/j.jmrt.2021.10.086>

- [10] A Bolouri, C G Kang. Correlation between solid fraction and tensile properties of semisolid RAP processed aluminum alloys, *J Alloy Compd.* 516 (2012) 192-200.
<https://doi.org/10.1016/j.jallcom.2011.12.045>
- [11] B Shayegh Boroujeny, M R Ghashghaei, E Akbari. Effects of SIMA (Strain Induced Melt Activation) on microstructure and electrochemical behavior of Al-Zn-In sacrificial anodes, *J Alloy Compd.* 731 (2018) 354-63. <https://doi.org/10.1016/j.jallcom.2017.09.316>
- [12] W C Liu, Y Sun, L X Hu. Microstructural properties, thermodynamics and kinetics of Al-Si semi-solid billet fabricated by liquid phase reaction sintering, *J Alloy Compd.* 885 (2021) 160932. <https://doi.org/10.1016/j.jallcom.2021.160932>
- [13] W C Liu, L X Hu, et al. Microstructure evolution and mechanical properties of Al-8wt.%Si semi-solid billet fabricated by powder metallurgy liquid phase reaction sintering, *Materials Science & Engineering A.* 802 (2021) 140656. <https://doi.org/10.1016/j.msea.2020.140656>
- [14] W C Liu, L X Hu, et al. Cu strengthened Al-Si-Cu semi-solid billet fabricated by liquid phase reaction sintering, *Materials Characterization.* 188 (2022) 111925.
<https://doi.org/10.1016/j.matchar.2022.111925>
- [15] W C Liu, Preparation via liquid phase reaction sintering and thixoforming of semi-solid Al-Si alloy, Ph. D thesis, Harbin Institute of Technology, 2023