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Solid Phase Gradient Alloying Method via ShAPE

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U.S. DEPARTMENT OF ENERGY

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1.0 Background

Fig. 1. Pictures of normal steel and stainless steel alloyed with Cr, Ni

Alloying has been used to confer desirable properties to various metallic systems since the bronze age. For example, Ni, Mn, and Cr are alloyed into an Fe matrix to make steel, which has improved strength and corrosion resistance (as displayed in Fig. 1). In addition to the type of alloying elements, the amount of alloying elements added into a matrix is also very critical for a material's mechanical property, physical property, machinability, and cost. Conventionally, new alloys are discovered by combining alloy precursors and elemental constituents into a mixture and melting them together. The solidified product or ingot represents a usually non-homogeneous combination of the chemistry with a microstructure dictated by the physics of solidification. It is time and energy intensive to optimize the alloying amounts via the casting process and hence, economically not conducive to rapid alloy discovery. In addition, the cast microstructure seen in the as-cast ingot is often not the ideal, nor even desired structure for optimized performance. Cast materials often need further processing such as homogenization, annealing, and deformation work put in through rolling, forging, extruding, etc. to have favorable microstructures and properties. A faster method to discover new alloy combinations and evaluate them in their worked or processed form is needed.

Recently, Laser Engineered Net Shaping was applied to fabricate gradient compositional material for alloy designing [1]. However, the mechanical properties of this gradient alloy were poor due to the existence of impurities, oxidation and cracks that are inherent in the melt-solidification process. Because of these limitations, we propose to use a solid-phase process (ShAPE) to create bulk materials (extrudates) that vary in composition from one end of the extruded solid to the other. Various manufacturing methods for alloy design including conventional casting method, laser based combinatorial method and current solid phase gradient alloying method are displayed and compared in Fig. 2. ShAPE machine and a schematic of ShAPE process are displayed in Fig. 3. Starting material is processed by a rotating and plunging die to form an extrudate.

We are proposing through this methodology to invent a new, bulk-scale combinatorial technique. With this novel technique, alloying element can be dissolved into the matrix with a continued gradient without bulk melting. Formation of inter-metallics and defects originating from melt processing can be avoided. In addition, the ShAPE process creates the mixed alloy chemistry, and meanwhile subjects the material to severe plastic strain, which induces the favorable "worked" microstructure. Products from the ShAPE process can be tested in hardness directly providing a path to rapid evaluation of mechanical properties. The ability to create graded structures using friction stir processing (FSP) has been demonstrated [2][3], however using the ShAPE process we believe will lead to much faster and higher fidelity results. When combined with high throughput screening methods of physical, mechanical and microstructural property characterization, efficiency and accuracy of alloy design can be significantly improved.

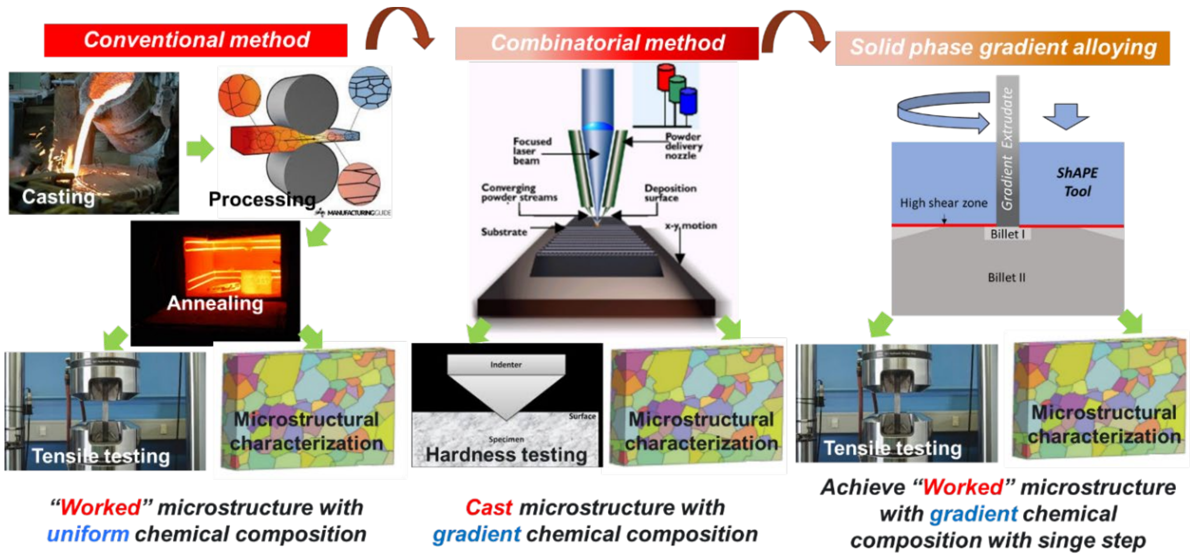

Fig. 2. Manufacturing methods for alloy design from left to right: conventional method based on casting; combinatorial method based on laser technique; and current solid phase gradient alloying method based on solid-state shear deformation.

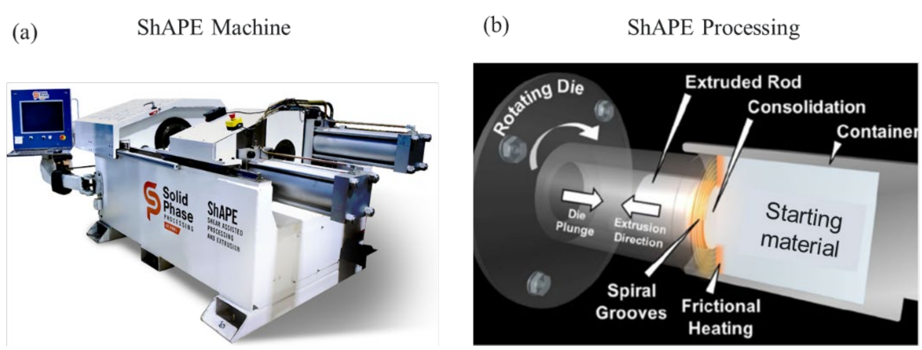

Fig. 3. (a) ShAPE machine and (b) schematic of ShAPE process to extrude a starting material to an extruded rod.

2.0 Specific Aims

Instead of studying shear deformation behavior of a fixed composition, the current method can investigate shear deformation behavior of a series of alloy compositions via one sample. The influence of alloying composition on shear deformation and the evolution of microstructure can be captured in a continuous way. This will help with a more complete description of the evolution of microstructure for SPPSi Thrust I, introducing a better methodology to observe the effects of chemistry. In addition, we can create data bases of shear deformation behavior for different alloy systems efficiently for SPPSi Thrust III, especially for efforts to generate insight from data analytic techniques that require large datasets. The dependence of the solubility of a second phase (or alloying element) on shear deformation and thermal cycles can be more easily illustrated, so that we might move towards the notion of a dynamic phase diagram for different alloy systems.

3.0 Materials and Methodology

Al-Si alloy system was selected to demonstrate the current solid-phase gradient alloying manufacturing method. Critical inputs including ShAPE parameters, billet design and tool design are investigated to ensure the controlled alloy compositional gradients. As displayed in Figs. 4 and 5, ShAPE tool and billet dimensions are designed in such a way that Al-4Si is processed first, then as the extrusion proceeds it is fed more and more from the underlying billet of pure aluminum. As the amount of pure Al being processed increases, a compositionally gradient Al-Si alloy will be obtained. The rate of dilution, alloy gradient obtained, and other aspects of the extrudate are set by features on the starting feedstocks, such as angle of the billet interface, design of the die face and ShAPE process parameters. Note that two different extrusion ratios (ERs) of 100 and 25 were applied in current project (Fig. 4). Meanwhile, ShAPE tools with flat die face and scrolled die face are both applied to investigate the influence.

After ShAPE process, optical microscopy (OM) and scanning electron microscopy (SEM) were used to quantify the distribution of Si particles through the whole gradient region to determine which billet design, ShAPE tool design, and ShAPE parameters are favorable to obtain a graded material. Additionally, microhardness were conducted on the longitudinal section of the ShAPE extrudate to investigate the correlation between the mechanical properties and the alloy gradient.

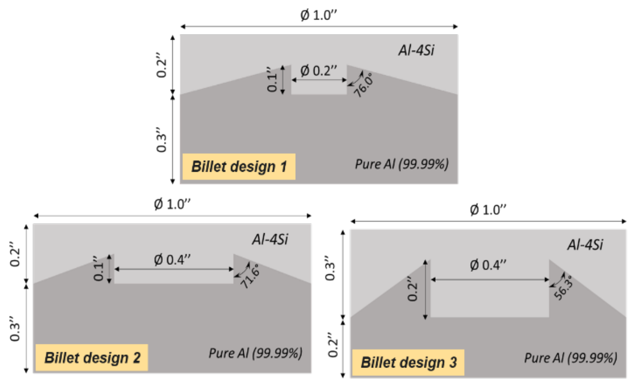

Fig. 5. Schematics of billet design to create gradient Al-Si material

4.0 Results

4.1 Effect of tool feature

Firstly, effect of ShAPE die face feature on Al-Si gradience is investigated, as displayed in Fig. 6. It shows that material mixing between Al-4Si and pure Al is poor using flat die (Fig. 6(a)) and tends to be more uniform with the scroll die (Fig. 6(b)). This is a common observation in solid phase processing that scroll feature on the tool can facilitate a more uniform material flow and result in a more homogenized microstructure. Therefore, ShAPE tool with scroll die feature is chosen for the following study.

In this comparison experiment between flat die or scroll die, extrusion ratio of the ShAPE tool is 100 and the extrusion hole diameter is 0.1 inch. Rotation rate of the ShAPE die is 300 RPM, while the ram speed is 4 mm/min. As for billet design, billet design 1 was selected.

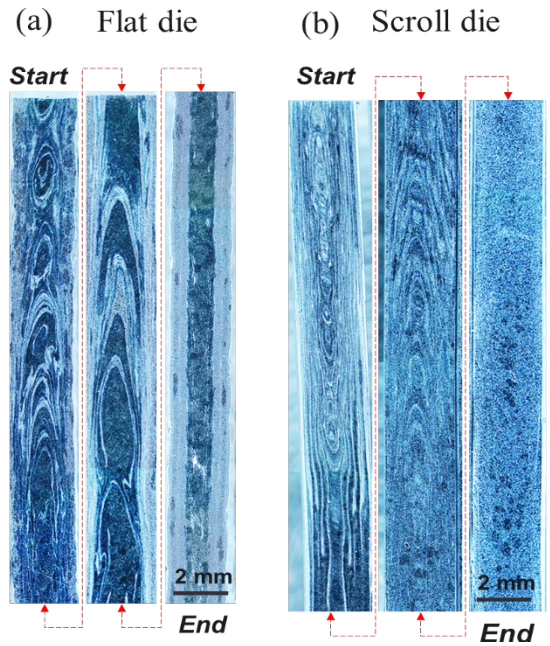

Fig. 6. Cross sections of extruded Al-Si via ShAPE with (a) flat die and (b) scroll

4.2 Effect of extrusion ratio

A ShAPE tool with extrusion hole diameter of 0.2 inch and extrusion ratio of 25 is used. The billet design 1, rotation rate of 300 RPM and ram speed of 4 mm/min were selected and to be the consistent with parameters in the section 4.1. Current section displays the effect of extrusion ratio on material mixing and resultant Al-Si gradience. As displayed in left OM of Fig. 7, Al-4Si system and pure Al are not mixed uniformly. A further observation via SEM on the selected regions shows that fine grains of Al-4Si exist in the extrudate start (Fig. 7(a)), and coarse grains of pure Al enter the material flow and form heterogenous microstructures as displayed in Figs. 7(b) and (c). In this case, material mixing and gradience are not ideal, and other critical inputs need to be modified.

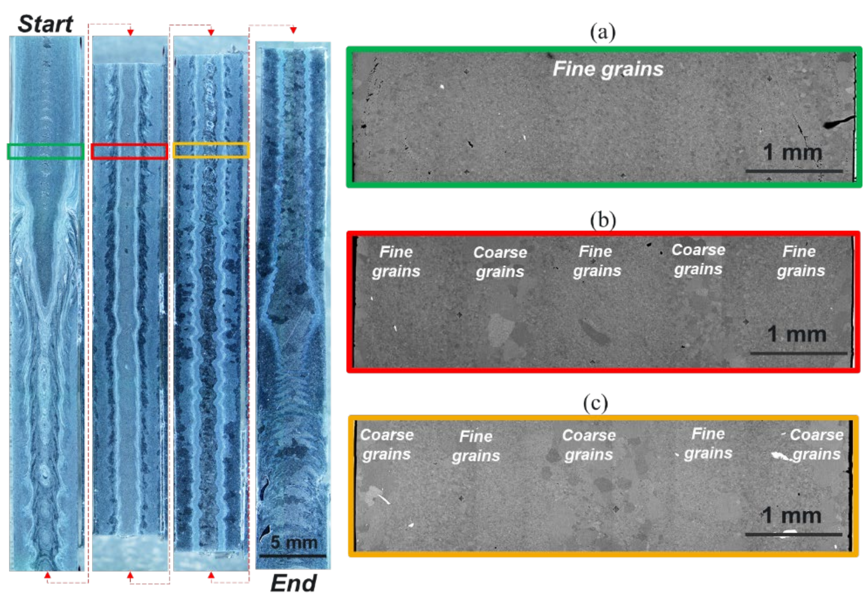

Fig. 7. Cross sections of extruded Al-Si via ShAPE with ER=25 and billet design 1.

4.3 Effect of billet design

The ShAPE tool of extrusion ratio of 25 is used. The billet design 2, rotation rate of 300 RPM and ram speed of 4 mm/min were selected. The gradient microstructure is obtained as displayed in Fig. 8 (left image), the Al-4Si and pure Al are mixed uniformly. A further observation via SEM on the selected regions shows that Si particle is uniformly distributed, and the content is decreased gradually (Figs. 8(a-c)). Meanwhile, an evident dependence of grain size on Si particle content is presented. Most of the Si particle exist on the grain boundaries. Detailed correlation between Si content and microstructure and local hardness will be discussed in section 4.6.

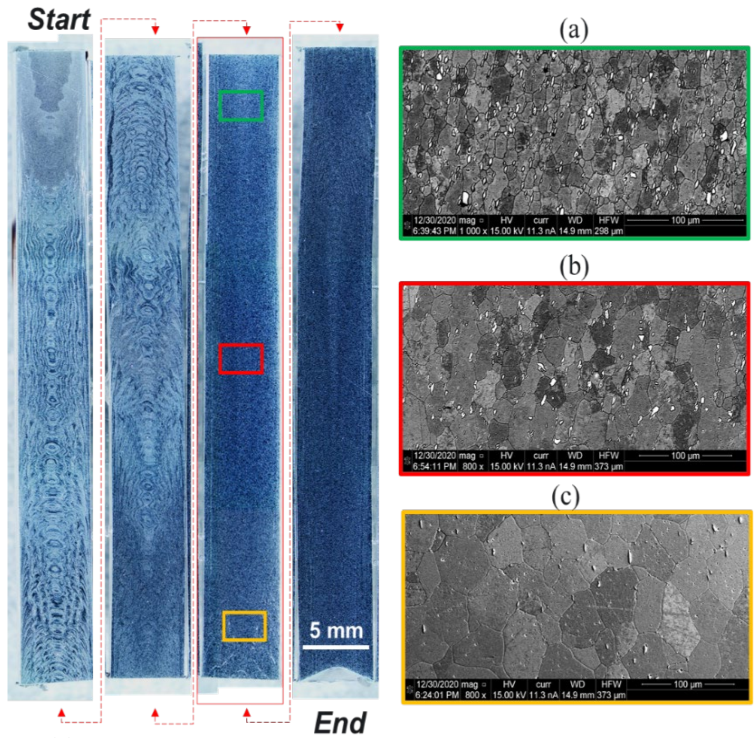

Fig. 8. Cross sections of extruded Al-Si via ShAPE with ... *25*

4.4 Effect of rotation rates

The ShAPE tool of extrusion ratio of 25 is used. The billet design 3 and a constant ram speed of 4 mm/min were selected. Two ration rates of 300 and 400 RPM were selected to investigate the influence of rotation rate on material flow and resultant gradience. As displayed in Fig. 9, the Al-4Si and pure Al are mixed uniformly when rotation rate is 300 RPM, while not uniformly when rotation rate is 400 RPM. Although the material mixing is uniform with billet design 3 and rotation rate of 300 RPM. Pores are observed in the extrudate. This can be attributed to the increased interfacial area between two billets (see Figure 5).

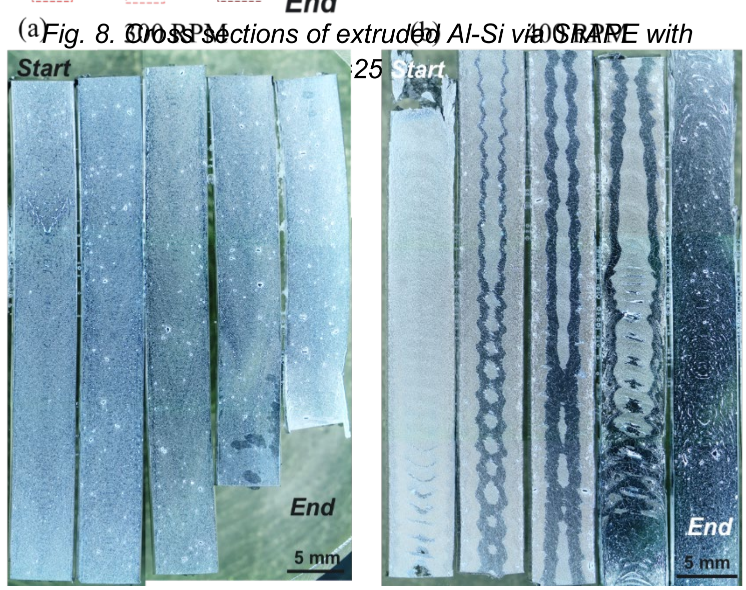

Fig. 9. Cross sections of extruded Al-Si via ShAPE with rotation rates of (a) 300 RPM and (b) 400 RPM.

4.5 Short summary

In this section, influence of tool feature, extrusion ratio, billet design and rotation rate on the material flow and resultant gradience is summarized in the Fig. 10. All the factors mentioned above determine the material gradience together. Therefore, we selected extrusion ratio of 25, billet design 2, and rotation rate of 300 RPM for our further analysis.

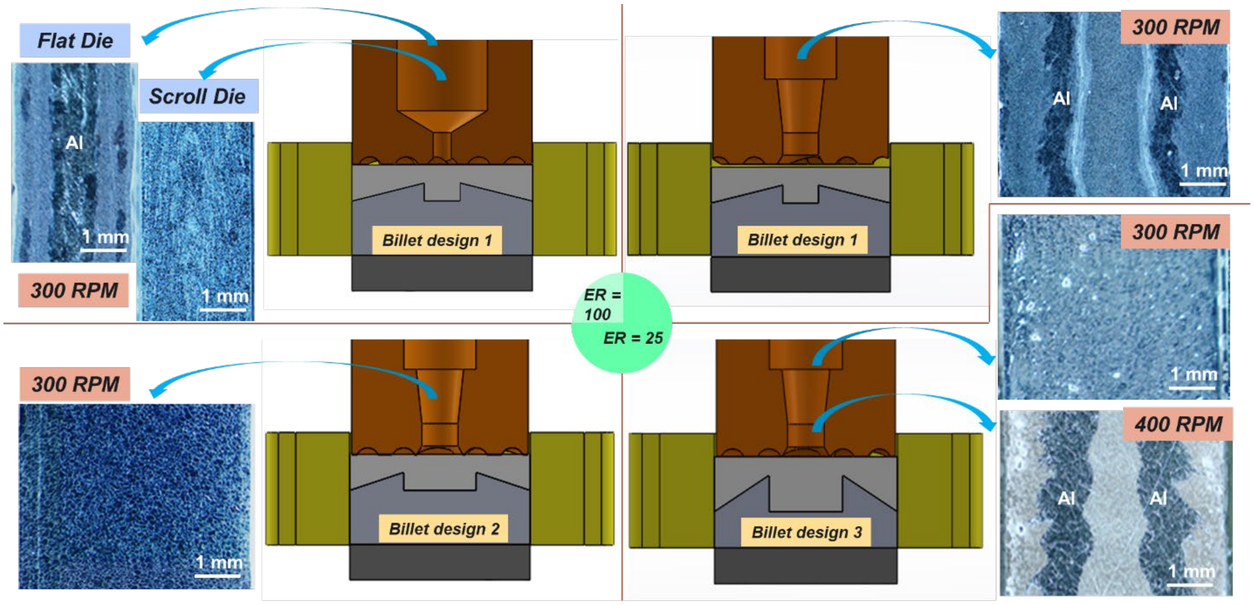

Fig. 10. Effect of tool feature, extrusion ratio, billet design and rotation rate on material gradience in Al-Si system.

4.6 Correlation between microstructure and hardness for Al-Si gradient material

As displayed in Fig. 11, the hardness gradually increases from low Si end towards high Si end. To quantify the Si content from low Si end to high Si end, SEM images were taken from one end to the other, as displayed in Fig. 12. With magnification of 1000, Si content can be determined as: 1.5 wt% to 6.8 wt%. It displays that the grain size decreases as the Si content increases. Furthermore, grain size distribution is presented in a larger scale with the magnification of 150.

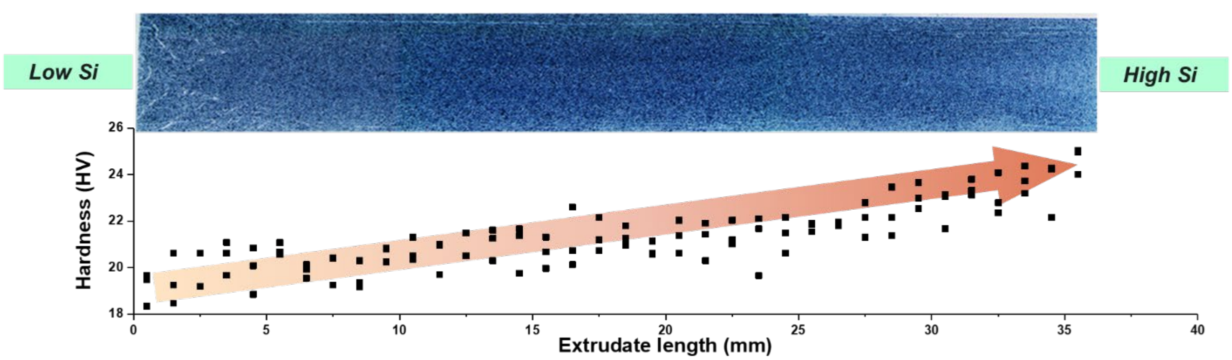

Fig. 11. Hardness along the gradient Al-Si material

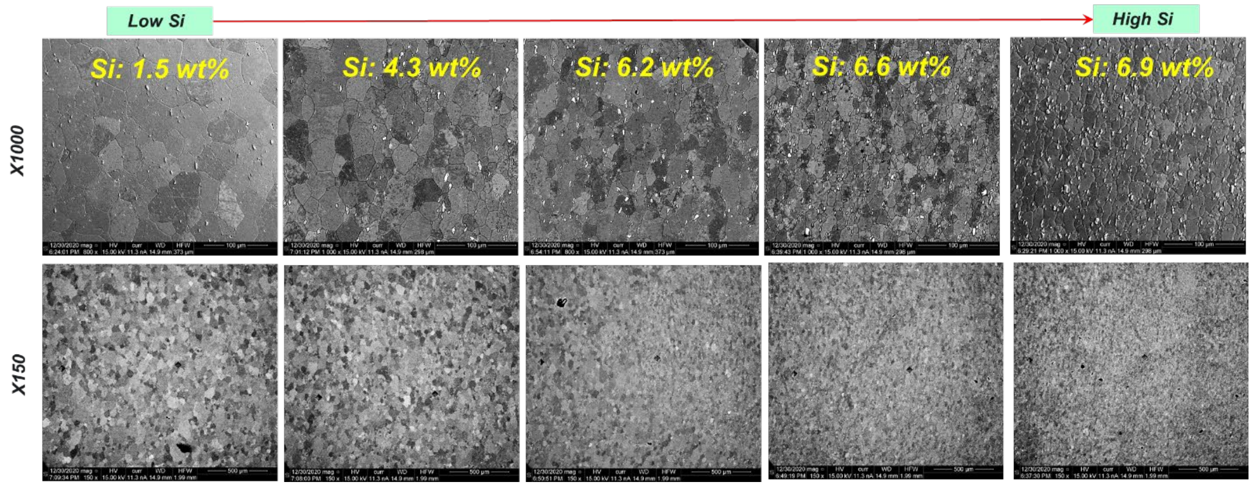

Fig. 12. SEM images from low Si content to high Si content in two magnifications: X150 and X1000.

5.0 Discussion

5.1 Heterogenous microstructure of Al-Si alloy

During exploring the proper billet design and ShAPE parameters, some interesting heterogenous microstructures were obtained, as displayed in Figs. 13(a-c). In these cases, material mixing between two billets is not sufficient and multiple layered heterogenous can be obtained. In recent years, heterogenous microstructure attracts a great amount of attention in academia due to their outstanding mechanical properties, and our solid phase alloying method might be an option for producing such microstructures.

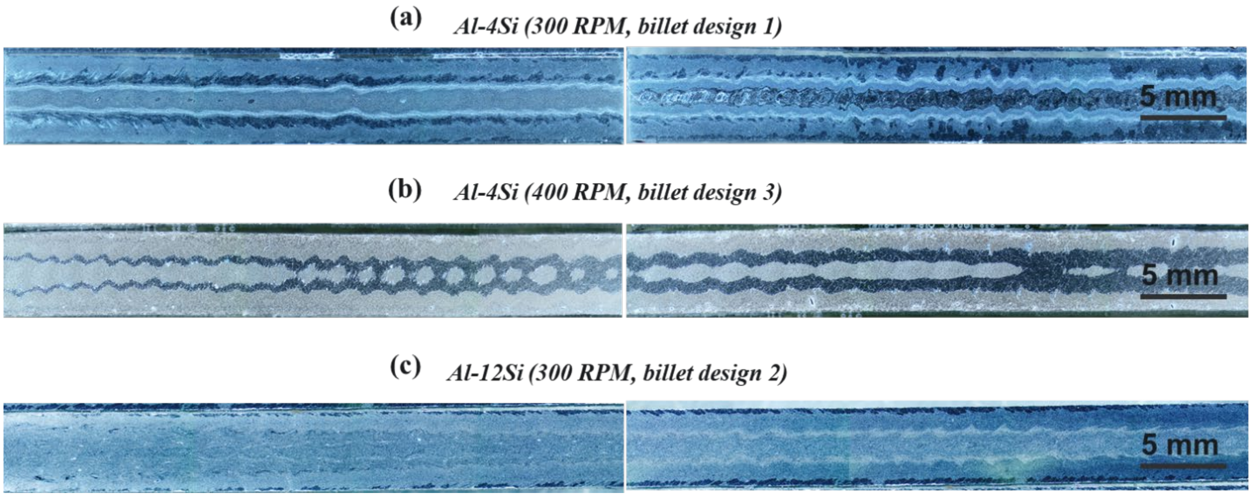

Fig. 13. Heterogeneous microstructure obtained with certain billet design and rotation rates.

5.2 Comparison between heterogenous microstructure and gradient microstructure

When the material mixing between two billet is sufficient, the Si partilces distribute within the extrudate in a gradually increased manner (right image of Fig. 14). On the other hand, poor mixing between two billet material would lead to heterogenous microstructure, namely, two material flow features maintrain their original Si content (left image of Fig. 14). Additiaonally, it can be concluced that the grain size is significanly influenced by the Si content.

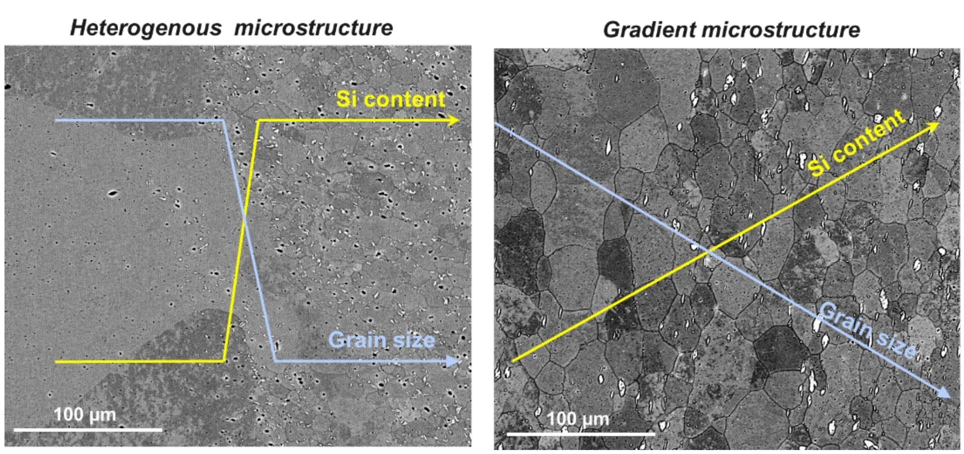

Fig. 14. Comparison between heterogenous and gradient microstructure.

6.0 6. Conclusions

A gradient-microstructure material of Al-Si alloy is fabricated from two billets (one is pure Al and the other is Al-Si alloy) via ShAPE in solid phase. The main conclusions can be summarized as below:
(1) The material gradience is determined by a series of factors including tool feature, extrusion ratio, billet design and rotation rate.
(2) In Al-Si alloy system, grain size decreases with the Si particle increasing.
(3) The hardness of Al-Si gradient material increases with the Si particle increasing.

7.0 7. Further directions

(1) The current solid phase gradient alloying method can be used for exploring other metallic material systems.
(2) Current method can create data bases of shear deformation behavior for different alloy systems efficiently.
(3) Current method can be extended to produce heterogenous-microstructure materials with favorable properties.

8.0 References

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