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Microstructure and mechanical properties of a novel rapidly solidified, high-temperature Al-alloy

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ABSTRACT

Rapid solidification (RS) processing, as a production method, offers a variety of unique properties based on far-from-equilibrium microstructures obtained through rapid cooling rates. In this study, we seek to investigate the microstructures and properties of a novel Al-alloy specifically designed for high temperature mechanical stability. Synthesis of, $AlFe_{11.4}Si_{1.8}V_{1.6}Mn_{0.9}$ (wt.%), was performed by two approaches: rotating cup atomization ("shot") and melt spinning ("flake"). These methods were chosen because of their ability to produce alloys with tailored microstructures due to their inherent differences in cooling rate. The as-solidified precursor materials were microstructurally characterized with electron microscopy. The results show that the higher cooling rate flake material exhibited the formation of nanocrystalline regions as well additional phase morphologies not seen in the shot material. Secondary dendritic branching in the flake material was on the order of 0.1–0.25 μm whereas branching in the shot material was 0.5–1.0 μm.

Consolidated and extruded material from both precursor materials was mechanically evaluated at both ambient and high (300 °C) temperature. The consolidated RS flake material is shown to exhibit higher strengths than the shot material. The ultimate tensile strength of the melt spun flake was reported as 544.2 MPa at room temperature and 298.0 MPa at 300 °C. These results forecast the ability to design alloys and processing approaches with unique non-equilibrium microstructures with robust mechanical properties at elevated temperatures.

1. Introduction

High temperature aluminum alloys offer a number of potential benefits that can be translated into both cost and weight savings over traditional materials such as titanium and nickel based alloys. The automotive industry in particular has invested considerably in alloy development by funding laboratory and production scale research to develop aluminum alloys that can exhibit enhanced high temperature (>300 °C) strength at low cost [1–3]. The need for lightweight high temperature materials is two-fold. Firstly, high performance engine components constantly undergo loading and unloading cycles that necessitate high temperature tensile and fatigue strength. Secondly, producing aluminum alloys with higher strength to weight ratios translates into direct mass reduction of engine components [4,5]. This leads to higher engine efficiency, decreased fuel consumption and reduced environmental impact [3,6–8].

Traditionally, common approaches to aluminum alloying have focused on increasing strength using three primary routes; solid solution strengthening, precipitation strengthening and cold/hot working. Of the three methods discussed, precipitation strengthened 2xxx, 6xxx and 7xxx alloys make up the majority of high strength aluminum materials used in automotive/aircraft applications [2,5,8]. Despite their high strength to weight ratios, the application of these materials is limited to operational temperatures below 100 °C–200 °C [9]. In the case of conventional precipitation strengthening, exceeding these temperatures results in an overaged material where coarsening of precipitate dispersions leads to an overall reduction in strength, and similar losses are observed in worked material due to microstructural recovery and possible recrystallization. In an effort to increase alloy strength at elevated temperature (~300 °C in service [2,7]), solid solution strengthening using solutes with limited diffusion at elevated temperatures presents a tractable choice in the development of new alloys.

The strength increases that can be obtained by solid solution strengthening during equilibrium solidification can be further improved using a far from equilibrium processing method such as rapid solidification (RS) [8,10]. Fast cooling rates effectively supersaturate the solid solution, through extension of solid solubility limits [2,6,8,10–12]. Diffusion and migration are severely limited at cooling rates greater than 10^3 K/s allowing the formation of non-equilibrium phases that are disordered or ultra-refined and that display enhanced compositional flexibility [3,11]. Incorporation of elements such as Fe, Si and Mn that have low diffusivity in aluminum enhances the thermal stability at higher temperatures [2,7,13]. Microstructures that develop in rapidly solidified materials tend to

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exhibit non-equilibrium microstructures, including amorphous, nanocrystalline or quasicrystalline phases [2,3,8,14–17]. As a result, grain size strengthening may further assist in increasing the yield point in these materials. RS alloys have also demonstrated improvements in mechanical properties such as ductility as well as fatigue and crack propagation resistance [7].

In this study, the microstructures and mechanical properties of a novel Al-alloy designed for high temperature applications are investigated. Specifically, far-from-equilibrium microstructures are synthesized via two rapid solidification approaches, each with inherently different cooling rates. These alloys are consolidated and mechanically tested at ambient temperatures and at 300 °C. The results show that the faster cooling rate promoted by melt spinning offers finer microstructures than those produced via rotating cup atomization. The benefits of the finer microstructure are demonstrated by enhanced low and high temperature mechanical properties in the consolidated, bulk state. Furthermore, the microstructures demonstrate thermal stability against coarsening in the as-consolidated state, indicated the ability to design future materials with enhanced mechanical properties in high temperature applications.

2. Material and methods

All testing was performed on a newly-developed alloy chemistry (AFM-11), the composition of which is shown in Table 1.

Rapid solidification (RS) of the Al–Fe high temperature alloy was conducted by two different methods: melt spinning of molten aluminum to produce flake material, and rotating cup atomization to form shot material. The melt spinning method utilizes a rapidly spinning, internally cooled copper wheel. As molten metal impinges on the surface of this cooled wheel, the stream is rapidly thinned during cooling, resulting in the formation of a metallic ribbon or flake material [18]. The rotating cup processing method is similar, however, instead of a cooled copper wheel, the molten metal is directed onto a rapidly spinning cup. As metal reaches the cup, centrifugal force disperses it into fine droplets (or shot) that cool convectively; high velocity gases are commonly employed to aid the cooling process [19]. Production of these materials was performed in an argon atmosphere. The cooling rate differences between these two methods are known to differ by several orders of magnitude and have been estimated using the secondary dendrite arm spacing.

Following fabrication of the RS flake or shot, these feedstock materials were subsequently canned, cold compacted and consolidated by vacuum hot pressing at 450 °C to ~90% theoretical density to form billets. The consolidated billets of both materials were then extruded at elevated temperature (~480 °C). Some additional melt spun flake material underwent an additional forging step (~480 °C). Identical pressing and extrusion parameters were used for both materials. The billet diameter was 50 mm and extrusions were produced using a 19:1 ratio, with a final diameter of 11.4 mm.

After extrusion, a minimum of three tensile specimens were produced. Tensile specimens were machined according to ASTM E8/E8M-09 and tested with the use of an Instron MTS 8800 tension tester. Sample sets were evaluated at both room temperature and elevated temperatures (300 °C) with a crosshead speed of 0.005 in./min. Crosshead displacement as well as extension of the specimen gage was measured.

Polished cross sections of the material were then fabricated and evaluated with a JEOL 7600 FESEM. Microstructural analysis and energy dispersive x-ray spectroscopy (EDS) examination of the specimens were performed both parallel and perpendicular to the extrusion direction to evaluate microstructural features and morphologies of the produced material.

Table 1
AMF-11 alloy composition (in wt.%).

Alloy	Fe	Si	V	Mn	Al
AFM-11	11.4	1.8	1.6	0.9	Bal.

3. Results & discussion

3.1. Rapidly solidified shot precursor material

In the 'as-solidified' state significant structural differences were observed between the rapidly solidified shot and flake material. This variation was expected due to the significant cooling rate differences between the two production methods. Both the melt spinning and rotating cup processing generated materials with a wide array of microstructures, differing primarily in length scale. As an example, secondary dendritic branching in the flake material was on the order of 0.10–0.25 μm whereas branching in the shot material was 0.5–1.0 μm. As seen in Fig. 1, the shape of shot is similar to a water droplet with elongation in the direction of the centrifugal force. The larger microstructural features of the shot material can be easily understood due to the comparatively slower cooling rates associated with this processing method over the melt spinning technique.

EDS data was collected from each of the different shot material morphologies and is presented in Fig. 2. The matrix phase consists primarily of aluminum. Dispersed between the rosette structures in Fig. 1c are areas of weak backscatter contrast that correspond to silicon enrichment (Fig. 2). The composition of these regions is approximately 70 wt.% Al and 30 wt.% Si, compositions were identified by using local area analysis of the EDS spectra.

EDS sum spectra of the entire mapped region were overlaid and are shown in Fig. 3. The dendritic phase was shown to have the highest iron content, followed by the rosette structure. The block-like, angular phase had the lowest iron concentration as well as the lowest Si concentration.

The decrease in both iron and silicon solubility is likely a result of cooling rate variation in the produced shot material. The increased solubility of iron and silicon in the dendritic structure indicates this morphology formed as a result of a faster cooling rate, followed by the rosette structure and finally the blocky/angular morphology. A significant body of literature has been devoted to estimating cooling rates in RS materials through measurement of the secondary dendrite arm spacing. Fig. 4 below shows secondary dendrite arm spacing that was measured and used to calculate an average spacing of 0.72 μm ± 0.2 μm.

While exact calculation of the cooling rate is dependent on diffusion coefficients and therefore alloy composition, an approximation of the cooling rate for aluminum alloys based on the work of Dobatkin et al. [20] indicates the cooling rate for the shot material is likely in the range 10^4–10^6 K/s.

3.2. Rapidly solidified flake precursor material

The melt spinning process utilizes a spinning copper wheel to produce the RS flake. The wheel is scribed with a special knurled pattern of lines, running both in the direction of rotation and perpendicular to it. This surface pattern produces discontinuous flakes that are approximately 1 mm × 1 mm square, with a thickness of 35 μm to 55 μm, depending on the operating parameters and materials. A micrograph of the cross-section of an Al–Fe RS flake is shown in Fig. 5. Nanocrystalline regions are present near the wheel side of the flake. The thickness of these nanocrystalline regions is variable, and can be attributed to large thermal gradients as well as the surface variation (knurled pattern) on the spinning copper wheel.

Aside from the nanocrystalline area, an additional bright circular phase was also visible (Fig. 5). EDS results indicated this phase was iron rich. The visible increase in diameter of this phase away from the wheel side of the flake is indicative of diffusion controlled growth and a cooling rate gradient through the thickness of the flake. Several other unique dispersed phases were identified in the AFM-11 material