Effect of different production methods on the mechanical and microstructural properties of hypereutectic Al-Si alloys

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Abstract

In this study, effects of different production methods i.e. melt spinning, high energy ball milling and melt spinning + high energy ball milling, on the mechanical and microstructural properties of hypereutectic Al-20Si-5Fe alloys were investigated. While Microstructural and spectroscopic analyses were performed using scanning electron microscopy and X-ray diffractometry, mechanical properties were researched using a depth-sensing indentation instrument with a Berkovich tip. Microstructural and spectroscopic analyses demonstrate that high energy ball milling process applied on the melt spun Al-20-Si-5Fe alloy for 10 min brings about a reduction in the sizes of silicon particles and intermetallics compounds.
However, further increasing milling time does not yield any significance reduction in the size of both silicon and intermetallics. High energy ball milling for 10 minutes on the starting powders is not enough to form any intermetallic phase. According to depth sensing indentation experiments, high energy milling of melt spun Al-20Si-5Fe alloys brings about an increment in the hardness values. For the present investigated Al-20Si-5Fe alloys, the production technique remarkably influences their elastic-plastic response to the indentation process in terms of both magnitude and shape of $P-h$ curves.

Keywords: powder metallurgy, mechanical alloying, rapid-solidification, microstructure, mechanical properties, elasticity

Introduction

The hypereutectic Al–Si alloys containing transition metals (TM) are among the structural materials used for high temperature applications. Compositional modifications of hypereutectic Al–Si alloys made by adding Fe, Co, Cu, and Ni improve mechanical properties at both room and elevated temperature [1-3]. What makes TM-containing Al-Si alloys durable at elevated temperatures is TM-containing intermetallic compounds dispersed in the microstructure [4]. Of the transition metals, Fe, perhaps, is the most important element for Al-Si alloys because it cannot be economically removed from the molten aluminum [2]. However, in traditional cast Al-Si-Fe alloys, Fe-bearing intermetallic compounds have a tendency to be coarse and needle-like morphology which is harmful for mechanical properties because of the stress concentration produced at the tip of the acicular phase during deformation process. Moreover, there is a big difference in deformation performance between
Fe-bearing phase and the Al matrix, which also has a detrimental effect on the mechanical properties [5,6]. In the cast Al-Si-Fe alloys, another subject that should be taken into account is coarse primary and eutectic silicon phases which deteriorate mechanical properties, as well. Actually, in all kinds of Al-Si alloys, the size and morphology of primary and eutectic silicon phases have vital importance in terms of mechanical properties. While higher volume fraction of silicon enhances the tensile strength of the alloy, large silicon particles in aluminum matrix can deteriorate the ductility of the alloy. Accordingly, modification of primary and eutectic Si, and neutralization of detrimental effects of Fe-bearing intermetallics in hypereutectic Al–Si-Fe alloys have been studied extensively to ensure adequate mechanical or tribological properties [7 -10]. Modification of the silicon particles and Fe-bearing phases can be achieved by different fabrication techniques such as spray deposition, gas atomization and melt-spinning [11- 15]. For example, by using spray deposition method, DISPAL series hypereutectic Al-Si-Fe alloys, which are already in commercial use, can be successfully produced [16]. Among the different fabrication methods, techniques of melt-spinning and high energy ball milling are two effective methods in terms of refining and modification of coarse silicon particles and acicular Fe-containing intermetallic phases [17, 18]. However, according to the best of our knowledge, there is so little or no information about the Al-20Si-5Fe alloys produced by a combination of these two methods, that is, melt spinning and subsequently high energy ball milling.

As it well known, hardness and elastic modulus have been two important mechanical properties of structural materials. Dept sensing indentation method allows determining elastic modulus \(E\), hardness \(H\) and stiffness \(S\), and also gives information about other elastic and plastic properties such as elastic and plastic deformations [19]. However, in terms of
mechanical properties, most of the data about hypereutectic rapidly solidified Al-Si alloys are about conventional microhardness data which does not contain information about elastic properties. Again, according to the best of our knowledge, there are very few or no reports about elastic properties of hypereutectic Al-20Si-5Fe alloys produced melt spinning (MS), high energy ball milling (HEBM). Therefore in this study, we produced Al-20Si-5Fe alloys by three different ways (HEBM, MS, MS+HEBM), and investigated mechanical properties of them by depth sensing indentation. We investigated the effects of different production techniques (HEBM, MS, MS+HEBM) on the microstructure and elastic-plastic properties of Al-20Si-5Fe alloys.

Theoretical Background

Figure 1 shows schematic illustration of indentation load \( P \) versus penetration depth \( h \) data obtained during one full cycle of loading and unloading process. The important parameters are peak load \( P_{\text{max}} \), the maximum depth \( h_{\text{max}} \), the final or residual depth after unloading \( h_t \), contact depth \( h_c \). Another important parameter is the slope of the upper portion of the unloading curve is \( S = dP/dh \) which is known as contact stiffness. Two important mechanical properties, the hardness \( H \) and elastic modulus \( E \) can be obtained from the loading and unloading data using following relations.

\[
H = \frac{P_{\text{max}}}{A}, \quad A = 26.43h_c^2
\]

(1)

where \( A \) is the contact area at the given load and


\[ E = \frac{s}{2} \sqrt{\frac{\pi}{A}} \]  

where \( E \) is the elastic modulus.

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**Material and Methods**

Pure Al powder with purity of 99.999\% (average particle size 106–250 μm), Si scrap with purity of 99.99\% (average particle size 3–5 μm) and Fe powder with purity of 99.99\% (average particle size 10–15 μm) were used as the starting materials in this study. A planetary ball mill (P100, TMC) with stainless steel vessel was used for high energy mechanical milling of the mixture in Ar atmosphere. The powders were milled at 800 rpm of rotating speed. Ball-to-powder weight ratio was 20:1, with stearic acid added at 10\% of the powder weight to moderate the cold welding process. In this study, all percentages are wt. \% unless otherwise stated. An Edmund Buhler SC melt-spinner, in which the molten alloy in a boron-nitride crucible (BN) was ejected onto a polished copper wheel, was used for production of rapidly solidified samples. The melt spinning processes were performed using a copper wheel with 20 m/s disc velocity by pressurized argon of 250 mbar. Melt-spun and high energy ball milled alloys were denoted as MS and HEBM, respectively. And the numbers 10 and 210 following HEBM denote the milling time of the alloys. That is, HEBM10 and HEBM210 denote the samples milled for 10 and 210 min, respectively. And MS+HEBM10 and MS+HEBM210 denote melt spun ribbons milled for 10 and 210 min respectively. In this study, milling time was selected 10 min for powder materials because of the fact that in the high energy ball milling process, the level of contamination will increase with increase of milling time and some undesirable phases may form if a powder is milled too long [20]. On the other hand, melt spun ribbons were subjected to different milling times such as 15, 20, 30, 60 and 210
because we want to see effect of high energy milling on the morphology and size of silicon and intermetallic phase. However, it was seen from optical and SEM micrographs, more than 10 min milling did not cause any remarkable change and reduction in the morphology and size of the silicon and intermetallic phases. That is why; the data related to milling of melt spun ribbons for 15, 20, 30 and 60 are not given here. All of the samples produced are shown in Table 1. The cross-sections of ribbons were selected for the indentation and scanning electron microscopy measurements. The microstructure and phase identification of the powder particles and melt spun ribbons were investigated by as Tescan MIRA LMH scanning electron microscopy with energy dispersive spectrometry, a DMAX2000 X-ray diffractometer with high energy monochromatic CuKα radiation (0.15418 nm) in the 2θ range of 15–80° at a scan rate of 0.05°/s. Indentation experiments were performed with a depth-sensing indentation instrument with a Berkovich tip (Shimadzu, DUH-W201S). All tests were performed under the same operating conditions. For all samples, the maximum load was 50 mN and the loading rate was 23.4 mN/s at each peak load.

Results and Discussion

Figure 2 shows the scanning electron microscopy micrographs of the samples with same composition produced by means of different production techniques. While a micrograph of conventional cast alloy has been given for comparison in Figure 2a, in Figure 2 (b, c, d and e) micrographs of samples produced by high energy ball milling for 10 minutes (HEBM10), melt spinning (MS), melt spinning + high energy ball milling for 10 minutes (MS+HEBM10) and melt spinning + high energy ball milling for 210 minutes (MS+HEBM10) have been illustrated, respectively. It is clearly seen that microstructure of conventional cast alloy is much coarser than those of other samples. In the samples MS, MS+HEBM10 and
MS+HEBM210, it was very difficult to distinguish primary and eutectic silicon phases because of their very small sizes. Except for the sample HEBM10 (Figure 2(b)), microstructures of all samples contain particle-like Si, acicular and plate-like intermetallics Figure 2 (c, d and e). In Figure 2 (b), powder particles of the sample HEBM10 are in irregular shape with size of ~ 120 μm. In the sample MS, average length/size of Si phases and intermetallics are 1.5 and 3.20 μm, respectively. In the sample MS+HEBM10, i.e. when high energy ball milling process was applied on the MS alloy, both average size of silicon and intermetallics decrease to 1.25 and 1.95 μm, respectively. However, further increasing the milling time did not bring about any significance reduction in the average size of both silicon and intermetallis, that is, average sizes of silicon and intermetallics were 1.20 and 1.94 μm, respectively, in the sample MS+HEBM210.

According to X-ray diffractometer results seen in Figure 3, acicular intermetallics in all samples are mainly δ-Al₄FeSi₂ phase, which is agreement with the results of ref. [21]. No other intermetallic phases could be detected in any of the alloys. Possible reasons include but not limited to: prevention of intermetallic phase formation such as β-Al₅FeSi by high cooling rate [22], and insufficiency of X-ray diffraction in detecting phases with low volume concentrations below 5 vol.% [21]. These results are quite compatible with our previous work [5]. It can also be concluded from Fig. 3 that except for the sample HEBM10, microstructures of all samples consist of Al, Si and δ-Al₄FeSi₂ phases. However, no intermetallic phase was determined in the sample HEBM10, meaning milling time was not enough to form an intermetallic phase [17].
Figure 4 shows peaks with the highest intensity of Si (111) and Al (111). In the Tables 2, intensities, FWHMs (full width at half maximum) and lattice parameters obtained from Al (111) peaks are given. The lattice parameter calculated for the sample conventional cast is well in agreement with the value of 0.4055 nm reported in literature [23]. According to (Figure 4 (a)), intensities of Si peaks of samples HEBM10, MS, MS+HEBM10 and MS+HEBM210 are 97.30, 52.27, 55.90 and 46.96 (a.u), respectively, which are smaller than the value of 158.59 for conventional cast sample. This may be attributed to solid solution of some of Si atoms in Al matrix because of extremely high cooling rate in melt spinning process, and it also seems that high energy ball milling process has a positive effect on the increment of solid solubility limits in Al-Si alloys. It is also clearly seen from Fig. 4 (b) and Table 2 that comparatively the sample conventional cast, Al (111) peaks generally shift to right in the samples HEBM10, MS, MS+HEBM10 and MS+HEBM210 (Figure 4 (b)) and the lattice parameters of the all samples are smaller than that of the sample conventional cast (Table 2) confirming melt spinning and high energy ball milling have positive effect on the solute solubility limits in Al-Si-Fe alloys. Because, Goldschmidt atomic radii of Si and Fe are 0.117 and 0.128 nm [24], respectively, which are smaller than the value of 0.143 nm for Al. That is why, with increasing solute solubility, it can be expected a decrease in the lattice parameter of the Al in Al-Si-Fe alloy. FWHMs of Al (111) peaks are seen as following arrangement (Table 2): HEBM10 < Conventional cast < MS ≅ MS+HEBM10 < MS+HEBM210 (Table 2). In literature, broadening of Al (111) peak is attributed reduction in the crystal size of Al phase [25]. Accordingly, in our study, Al (111) peaks of the samples MS, MS+HEBM10 and MS+HEBM210 are bigger than that of the sample conventional cast meaning MS and MS+HEBM processes causes reduction of sizes of α-Al phases. However, HEBM process for 10 min brought about a slight increase in the size of α-Al phase of the sample HEBM10 due
to agglomeration of powder particles at durations until 10 min in high energy ball milling process [17].

Microstructure of the sample HEBM10 is composed of only Al and Si phases. Figure 5 shows Scanning electron microscopy-Mapping analyses taken from the sample HEBM10. During high energy ball milling process in case of ductile–brittle powder mixtures, the brittle particles fractured and were trapped at the weld interfaces between ductile particles. The continued fracture and cold-welding processes resulted in a uniform distribution of the brittle particles in the ductile matrix [17]. In our case, brittle Fe and Si particles were uniformly dispersed/embedded in the Al matrix in a milled state as well (Figure 5).

The representative P-h curves recorded from dynamic microindentation of specimens HEBM10, MS, MS+HEBM10, MS+HEBM210 are given in Figure 6. It can be concluded from Figure 6 that, for the present investigated Al-20Si-5Fe alloys, the production technique remarkably influences their elastic-plastic response to the indentation process in terms of both magnitude and shape of P-h curves (Fig. 6). Slope of loading curve of the sample HEBM10 is very low compared to those of other three samples (Fig. 6). It is also clearly seen from Figure 6 that treating melt spun ribbon by high energy ball milling effects its elastic-plastic behavior: slope of the loading curve is increasing with increasing milling time, meaning increasing milling time enhanced the ability to strain hardening in small volume of the melt-spun ribbons. Changing the unloading parts to a higher penetration depth means that indented materials were subjected to higher plastic deformation. As it is well known, hardness is described as the resistance of materials against plastic deformation [19]. Therefore, considering loading-unloading data, hardness of the specimens can simply be arranged as HEBM10< MS< MS+HEBM10< MS+HEBM210. On the other hand, hardness values
calculated using Eq. (1), are also given in Table 3 and changes of them with different production method, which is consistent with the results above, are illustrated in Figure 8. According to Suryanarayana [26], during high energy milling, the powder particles are repeatedly flattened, cold welded, fractured and rewelded. The force of impact plastically deforms the powder particles leading to work hardening. Therefore, we think that the reason of the increasing in hardness values of high energy ball milled ribbons with increasing milling time is mainly work hardening mechanism.

Figure 9 shows changing of elastic modulus ($E$) with different production techniques. It is also seen from Table 3 that although, there is not any significant difference in the elastic modulus values of the samples, the lowest $E$ belongs to the sample HEBM10. As for melt spun ribbons, when high energy milling process is applied for 10 minutes, $E$ becomes maximum, but for further increasing milling time up to 210 minutes it decreases. It has been reported that with increase strain amplitude because of cycle fatigue loading, the averaged density of the dislocations in the sample tends to increase, resulting in an increase of the flow stress or the yield strength of the material and also Young’s modulus [27]. Therefore, elastic modulus of the sample MS+HEBM10 is higher than those of the samples HEBM10 and MS. However, further increasing milling time brings about decrease of the elastic modulus of the sample (MS+HEBM10), and this situation can be explained as the following: During mechanically milling/alloying, due to kinetic energy of the grinding media (balls), intense mechanical deformation increases the temperature of materials in the milling jar. Increase in the temperature during milling is mainly due to ball-to-ball, ball-to-powder, and ball–to–wall collisions and also due to frictional effects. Therefore, the higher grinding energy (milling speed, relative velocity of the balls, time of the milling, size of the grinding balls, etc.), the
higher is the temperature rise [20]. For example, Takacs and McHenry (2006) [28] reported that in same condiditon planetary milling operations, the ball temperature was measured below 100 °C and over 200 °C at 200 and 280 rpm milling speeds, respectively. It was also reported that in a planetary milling experiment, the ball temperature can be as high as 500 – 600 °C. On the other hand, Unlu et al. (2002) [29] reported that in the rapidly solidified Al-Si alloys, coarsening of Si phases starts around 348 °. Therefore, we can conclude that the increment in the temperature in the milling jar should be enough high to consider in terms of Si phases because of milling speed was 800 rpm which is quite higher than that of reported by ref [20, 28]. If the temperature experienced by the powder is high, the associated higher diffusivity (higher atomic mobility) leads to recovery of defects [20]. So, reduction in the elastic modulus of the sample MS+HEBM10 may be attributed to defect recovery because of increment of the temperature during milling.

According to the theory of work of indentation proposed by Stillwell and Tabor [30], the necessary work to form an indentation can be determined using loading and unloading data. The area under the loading curve equals to total work $W$ done by indenter during indentation experiment. While the reversible elastic work ($W_{E}$) can be deduced from the area under unloading curve, the energy absorbed by the plastic deformation alone equals to the difference between total work and elastic work ($W_{P}=W-W_{E}$) [31 - 33]. In the present study, as the maximum load ($P_{max}$=50mN) is the same for all samples, we can assume $h_{max}$, $h_{f}$, and $h_{max} – h_{f}$ as a measure of total work, plastic work and elastic work, respectively. Due to unloading from $h_{max}$ to $h_{f}$ is elastic, and $h_{max} – h_{f}$ is related to elastic depth, we can use $h_{max} – h_{f}$ as a parameter which represents elastic recovery rate. Figure 7 shows changing of elastic recovery rate ($h_{max} – h_{f}$) with different production techniques. And, values of elastic
recovery rate \((h_{max} - h_f)\) are listed in Table 3. It is clearly seen that the sample HEBM10 has the highest elastic recovery rate \((h_{max} - h_f)\) value. Other three samples’ elastic recovery rates are quite lower than that of the sample HEBM10. It is also concluded that increasing milling time brings about slightly enhancing elastic recovery rates of melt-spun ribbons.

Conclusion

In summary, present study reports the effect of different production ways on the microstructure and mechanical properties of hypereutectic Al–20Si–5Fe alloys. The conclusions are as follows:

- Microstructure of conventional cast alloy was much coarser than those of the samples produced by techniques of melt spinning, high energy ball milling, and melt spinning following by high energy ball milling.
- Melt spinning process brought about formation of finer silicon and intermetallics phases according to high energy ball milling for 10 min.
- When the melt spun Al-20Si-5Fe alloy was subjected to high energy ball milling for 10 min, there was a reduction in the sizes of silicon particles and intermetallics compounds of it. However, further increasing milling time did not bring about any significance reduction in the size of both silicon and intermetallis.
- Microhardness values of melt spun ribbons were higher than those of the samples produced by high energy ball milling. On the other hand, when the melt spun Al-20Si-5Fe ribbon was subjected to high energy ball milling, microhardness value of it also increased.
- For the present investigated Al-20Si-5Fe alloys, the production technique remarkably influences their elastic-plastic response to the indentation process in terms of both magnitude and shape of $P-h$ curves.

- No intermetallic phase was determined in the sample HEBM10; present meaning milling time was not enough to form an intermetallic phase and solid solution.

- In the present study, the lowest $E$ value belongs to the sample HEBM10. As for melt spun ribbons, when high energy milling process is applied for 10 minutes, $E$ becomes maximum, however, when milling time is further increased up to 210 minutes it decreases.

- The sample HEBM10 has the highest elastic recovery rate ($h_{max} - h_f$) value. Other three samples’ elastic recovery rates are quite lower than that of the sample HEBM10. It is also concluded that increasing milling time brings about slightly enhancing elastic recovery rates of melt-spun ribbons.

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References


Figure captions

Figure 1 Schematic illustration of indentation load ($P$) versus penetration depth ($h$) data obtained during one full cycle of loading and unloading process.

Figure 2 Scanning electron microscopy micrographs of Al-20Si-5Fe alloys produced by different production techniques; (a) conventional casting, (b) high energy ball milling for 10 min (HEBM), (c) Melt spinning (MS), (d) Melt spinning + high energy ball milling for 10 min (MS+HEBM10), (e) Melt spinning + high energy ball milling for 210 min (MS+HEBM210)

Figure 3 X-ray diffraction patterns of Al-20Si-5Fe alloys fabricated by different production methods.

Figure 4 X-ray diffraction patterns of Al-20Si-5Fe alloys showing the peaks with the highest intensity of (a) Si (111) and (b) Al (111)

Figure 5 Scanning microscopy -Mapping analyses from Al-20Si-5Fe alloy fabricated by high energy ball milling for 10 min.
Figure 6 The representative P-h curves recorded from a Berkovich dynamic microindentation of the specimens HEBM10, MS, MS+HEBM10, MS+HEBM210

Figure 7 Changes in the elastic recovery rate of Al-20Si-5Fe alloy by different production techniques

Figure 8 Changes in the microhardness value of Al-20Si-5Fe alloy by different production techniques

Figure 9 Changes in the elastic modulus value of Al-20Si-5Fe alloy by different production techniques
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Table 1 Chemical composition and production ways of the samples used in this research

<table>
<thead>
<tr>
<th>Sample</th>
<th>Si (wt%)</th>
<th>Fe (wt%)</th>
<th>Al (wt%)</th>
<th>Production way</th>
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<tr>
<td>HEBM10</td>
<td>20</td>
<td>5</td>
<td>bal.</td>
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<tr>
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<td>20</td>
<td>5</td>
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<tr>
<td>MS+HEBM10</td>
<td>20</td>
<td>5</td>
<td>bal.</td>
<td>Melt spinning subsequently high energy ball milling for 10 min</td>
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<tr>
<td>MS+HEBM210</td>
<td>20</td>
<td>5</td>
<td>bal.</td>
<td>Melt spinning subsequently high energy ball milling for 210 min</td>
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Table 2. Full width at half maximums height (FWHMs), Lattice parameters ($a$) of Al (111) peak

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<th>FWHM</th>
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<td>MS</td>
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<tr>
<td>MS+HEBM10</td>
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<tr>
<td>MS+HEBM210</td>
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<td>0,4055</td>
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Table 3. Microhardness ($H$), Elastic modulus ($E$) and Elastic recover rate ($h_{\text{max}}-h_{f}$), the maximum depth ($h_{\text{max}}$) and the residual depth values of the Al-20Si-5Fe alloys produced different production ways

<table>
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<th>$h_{\text{max}}$ ($\mu$m)</th>
<th>$h_{f}$ ($\mu$m)</th>
<th>$h_{\text{max}}-h_{f}$ ($\mu$m)</th>
<th>$H$ (MPa)</th>
<th>$E$ (GPa)</th>
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