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FABRICATION OF Fe-Mn-Si ALLOY BY MECHANICAL ALLOYING AND DIRECT CURRENT SINTERING

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ABSTRACT

The results of a study of Fe-Mn-Si system for shape memory applications are reported .Fe, Mn and Si elemental powders with chemical composition of $Fe_{60}Mn_{29}Si_{11}$ (at%) were mechanically alloyed for 30 hours by a planetary ball mill, compacted and sintered at 1027K. The composed phases were investigated and mechanical alloying repeatability was evaluated. The elemental powders formed into γ-FCC and ε-HCP phases after mechanical alloying, and eventually into γ-FCC phase after compacting and sintering, which indicates that the sample has possibility to show shape memory effect due to stress induced martensitic transformation. Furthermore, a presence of small amount of nano-sized phase was confirmed after mechanical alloying, which might influence the materials properties. It was observed that two different composed phases formed after MA for 20 hours even though the samples were produced at the same experimental condition settings but local manganese concentration and temperature difference might influence to this difference. It is suggested that mechanical alloying conditions have to be optimized.

Keywords: *Fe-Mn-Si Alloy, shape memory material, mechanical alloying, direct current sintering , XRD pattern.*

1.0 INTRODUCTION

Fe-Mn-Si alloy system shows shape memory effect due to stress induced martensitic transformation by stacking faults formation and the reverse transformation by heating. The martensitic transformation occurs between γ-face centered cubic (FCC) and ε-hexagonal close packed (HCP) phases which are iron-based solid solution, namely, manganese and silicon atoms substitute randomly the position of iron atom in α-body centered cubic (BCC) structure. The composition of the alloy system has been investigated and optimized by several research groups for recent years. There are several techniques to produce the Fe-Mn-Si shape memory alloy. The vacuum melting techniques have been extensively investigated. Mechanical alloying (MA) is one of techniques used to

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obtain alloyed powder. It has several advantages to produce unique microstructures, nanoscale structures and nonequilibrium phases like amorphous one, which lead to improve materials properties. Therefore, it is important to investigate the Fe-Mn-Si alloy system produced by MA. In this study, MA of Fe, Mn and Si elemental powders by a planetary ball mill, and their subsequent compacting and sintering was carried out. The composed phases were investigated and MA repeatability was evaluated.

2.0 EXPERIMENTAL PROCEDURE

Commercially pure Fe (99.9%), Mn (99.9%) and Si (99.9%) elemental powders with composition of $Fe_{60}Mn_{29}Si_{11}$ (at%) were mechanically alloyed in stainless steel vials (45ml) with several stainless steel balls. The atmosphere was argon gas (99.999%) in order to minimize the oxygen contamination during MA. The ball-to-powder weight ratio was approximately 8:1. The rotation speed was 600 rpm. The alloying period of 30 min was alternated with an equal rest time in order to avoid excessive increasing temperature during MA. The MA was performed by a Frisch Pulverisette 7 planetary ball mill. The powder after MA was compacted and sintered at 1027K for 10 mins under vacuum condition (2Pa) at a pressure of 40MPa by means of pressure assisted direct current sintering. The crystal phases of the samples after MA were determined by X-ray diffraction (XRD) equipment, a Siemens D5000 diffractmeter, using Cu-Kα radiation at 40kV and 45mA and a graphite monochromator at room temperature. The refinement of the XRD patterns was carried out with the *Fullprof* program. Scanning electron microscopy was performed by a Nova NanoSEM 200 at an accelerating voltage of 18kV to observe the morphology of powder after MA.

2.1 Experimental Apparatus

2.1.1 Mechanical Alloying (MA)

MA is one of the techniques used for production of alloyed powder with solid state reaction at high energy collisions between balls and powder particles. The process consists of cold welding, fracturing and rewelding of powder particles. Different types of MA equipments have been used to produce and investigate new materials by several research groups recently. In this study, a planetary ball mill equipment was used. It has advantage to generate high impact energy with approximately 20 times the gravitational acceleration. Figure 1 (left) shows schematic image of the planetary ball mill.

2.1.2 Pressure assisted direct current sintering

Direct current sintering is one of sintering methods applying high current, which offers many advantages, that is; the temperature can be controlled precisely, the sintering time is reduced and a high energy efficiency can be obtained, etc. On the other hand, pressure assisted sintering is the way of fabrication of desired shape of the final product, which can be produced directly from powder. The combination of pressure assisted sintering with direct current is one of the methods to obtain bulk sample with many advantages. Figure 1 (right) shows schematic image of pressure assisted direct current sintering set-up.

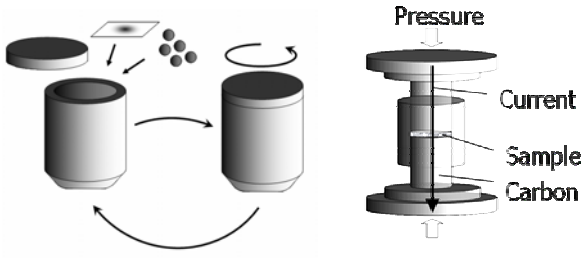

Figure 1: Schematic image of planetary ball mill (left) and pressure assisted direct current sintering set-up (right).

3.0 RESULTS AND DISCUSSION

Figure 2 shows XRD patterns for powder after several cycles of MA, showing that α-BCC phase persists at early stage of MA (5 hours). However, intensity of the peak decreases with increasing MA time and eventually the diffraction peaks of γ-FCC and ε-HCP phases are observed after MA for 30 hours. It indicates that manganese and silicon atoms diffused into α-BCC matrix, and subsequently, γ-FCC and ε-HCP phases formed after MA for 30 hours. This suggests that the sample after MA has possibility to show shape memory effect. The ε-HCP phase is martensitic phase appearing after stress induced martensitic transformation. The effect is attributed to stacking fault formation because of high energy collisions between balls and powder particles during MA. However, the XRD peak of the phase was broadened with increasing MA times, suggesting that a lot of lattice defects were produced with MA. In addition, it was noticed that two different results were obtained after MA for 20 hours, even though the samples were produced by the same experimental condition settings. Figure 3 and 4 show XRD patterns after MA for 20 hours for *Sample 1* and *Sample 2*. The refinement of the XRD patterns was carried out in order to analyze the results accurately with γ-FCC, ε-HCP and α-BCC structures whose space groups are the *Fm3m*, $P6_3/mmc$ and *Im3m* respectively. γ-FCC and ε-HCP phases were confirmed for both samples after MA for 20 hours. However, α-BCC phase also formed for *Sample2*. This means that *Sample 2* is still in the course of transformation, while *Sample 1* is already properly transformed. It can be assumed that MA for 20 hours is a key turning point to make the transformation at the MA conditions used in this study. Furthermore, the existence of α-BCC phase depends on manganese concentration and temperature according to Fe-Mn binary phase diagram in the range below 30 at% of manganese concentration. Therefore, the different result obtained is probably due to local manganese concentration and local temperature differences during MA. It is suggested that MA conditions have to be optimized carefully in order to get a reliable repeatability.

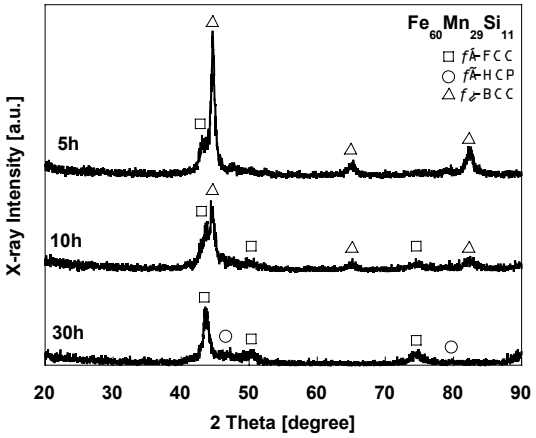

Figure 2: XRD patterns for a sample at various alloying times.

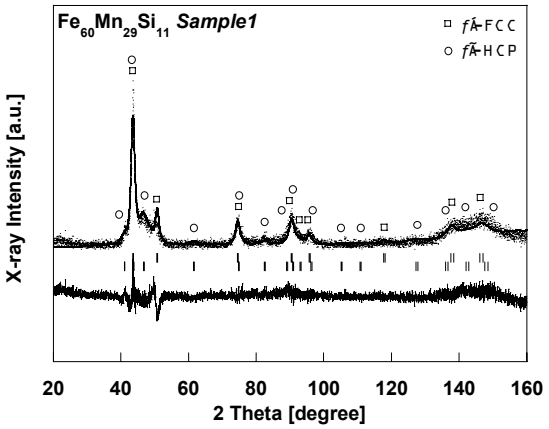

Figure 3: XRD pattern and its fitting results for *Sample 1* after MA for 20 hours.

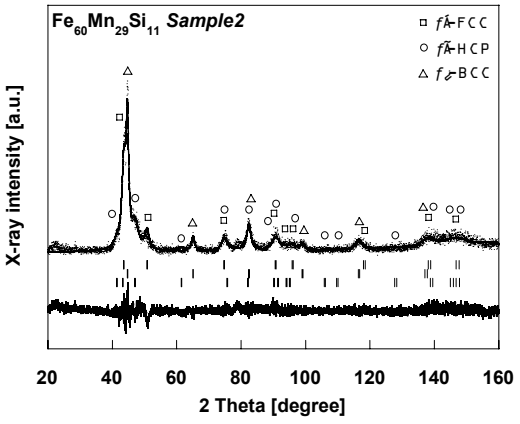

Figure 4: XRD pattern and its fitting results for *Sample 2* after MA for 20 hours.

The morphology of *Sample 1* and *Sample 2* after MA for 20 hours is shown in Figure 5 (a) and (b) respectively, and the magnified images of (a) and (b) are shown in Figure 5 (c) and (d), respectively. It is noticed that size of the particles for *Sample 1* was relatively smaller than that for *Sample 2*, which is one of the differences between *Sample 1* and *Sample 2* after MA for 20 hours. The mean diameter of the particles of *Sample 1* and *Sample 2* are about 40μm and 60μm, respectively. It can be noted that *Sample1* was better alloyed and milled than *Sample 2*, therefore, the size of powder particles for *Sample1* is smaller than that for *Sample 2*. Meanwhile, it is worth noting that nano-sized phase precipitation (dark spot) in the particle was confirmed for both *Sample 1* and *Sample 2*, Figure 6. The diameter of the phase is about 100nm, which might influence the materials properties.

The sample after MA for 30 hours was compacted and sintered by pressure assisted direct current sintering. Figure 7 shows XRD pattern for the sample after compacting and sintering at 1027K, showing clear diffraction peaks of γ-FCC phase. It indicates that the sample after a series of MA and sintering has possibility to show shape memory effect due to stress induced martensitic transformation.

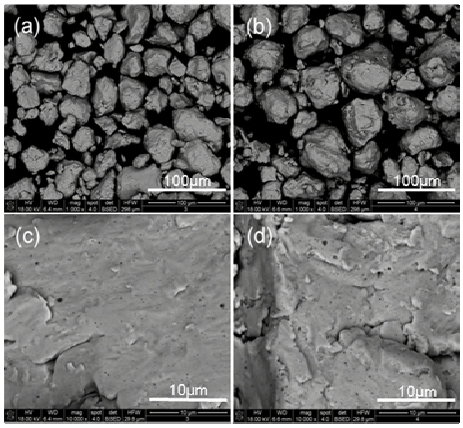

Figure 5: The surface configurations of (a) *Sample 1*, (b) *Sample 2* after MA for 20 hours, and those magnified are shown in (c) and (d), respectively.

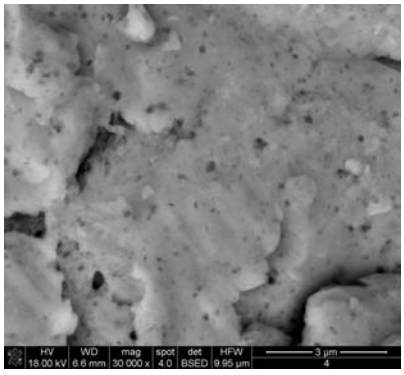

Figure 6: The magnified surface configuration for *Sample 1*.

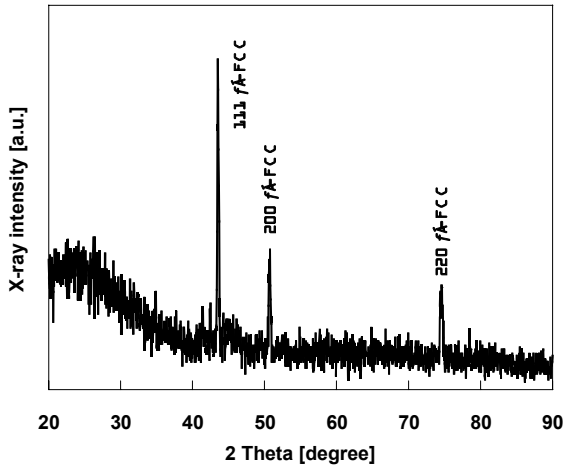

Figure 7: XRD pattern for sample after MA for 30 hours and compacting and sintering at 1027K.

4.0 CONCLUSIONS

Fe, Mn and Si elemental powders with composition of $Fe_{60}Mn_{29}Si_{11}$ were mechanically alloyed for 30 hours by a planetary ball mill, compacted and sintered at 1027K. The composed phases were investigated and mechanical alloying repeatability was evaluated. Results obtained are summarized as follows:

i. γ-FCC and ε-HCP phases formed after MA for 30 hours, suggesting that the sample after MA has possibility to show shape memory effect.
Furthermore, MA enables us to obtain ε-HCP phase owing to high energy collision during MA.

ii. Two different phase contents appeared after MA for 20 hours even though the samples were produced by the same experimental condition settings. This is probably due to local manganese concentration and temperature differences. It is suggested that mechanical alloying conditions have to be optimized.

iii. Nano-sized phase precipitations were observed for samples after MA, which might influence the materials properties. The diameter of the phase observed is about 100nm

iv. Clear diffraction peaks of γ-FCC phase were observed after compacting and sintering, suggesting that the sample after the series of processes involved in this fabrication processes has also possibility to show shape memory effect.

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