Transactions of the Materials Research Society of Japan 29 [8] 3499-3502 (2004)

Fine Grained Carbon Steel via Mechanical Alloying and Hot Pressing

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Grain refinement is known as the most effective way to improve mechanical properties of steel. In the present study, mechanical alloying (MA) and hot pressing (HP) were subjected to iron and graphite powders mixture with hypo-eutectoid composition to provide steel with fine structure. MA was carried out using a planetary ball mill for 100, 200 and 300 h, while HP at 41 MPa at various temperatures: below, near and above the eutectoid transformation point A_1. The mechanically alloyed (MAed) powders were characterized by X-ray diffraction (XRD), differential thermal analysis and scanning electron microscopy (SEM), while, the hot pressed (HPed) compacts by SEM, Vickers hardness measurement and tensile test. During MA, refinement of crystallite, formations of super-saturated iron solid solution and Fe/C amorphous phase occur first. With further MA time, these phases begin to transform to more stable phases such as carbides. In the case of HP at 610°C (below A_1), very fine cementites are precipitated in fine ferrite grain with sub-micron meter in the size. However, the good mechanical properties cannot be attained because of low sinterability. At 730°C (near A_1), the strength reaches the maximum value. With further temperature increase (at 800°C), the sintering progresses well and the coarsening occurs, resulting the decrease in the strength. However, the fracture strain increases significantly. The steels obtained in the present study have mechanical properties that are comparable to those of standard (JIS) steels through the well-established heat treatment such as normalizing and thermal refining.

Key words: Mechanical alloying, fine grain ferrite, fine cementite, hot pressing, heat treatment

1. INTRODUCTION

Mechanical properties of steels such as strength and toughness usually can be improved by grain refinement through thermal refining and addition of alloying elements. However, the grain refinement through thermal refining has a limitation up to only several ten micrometers. On the other hand, in ecomaterial perspective, the addition of alloying elements (such as special elements that become trumped elements etc.) is not desirable due to the problem in recycling process and resource depletion.

Recently, mechanical alloying (MA) was utilized to produce non-equilibrium phases such as amorphous [1,2], super-saturated solid solution [2] and nano-crystalline phases [3,4]. There were reported [5-8] that these phases also could be obtained in hyper-eutectoid Fe-C systems. The alloying mechanism during MA also had been discussed [9,10]. During MA, amorphous Fe/C phase and super-saturated iron solid solution transform to metastable carbides such as Fe_3C and Fe_7C_3 [9,11-13].

It is supposed that if the MAed Fe-C powders were consolidated, very fine-grained carbon steels could be attained. Kimura et al [7] and Nurul Taufiqu Rochman et all [8] have succeeded to produce Fe-high C system hard alloys from the MAed powders.

In order to make a comparative study, therefore, in the present study hypo-eutectoid Fe-C powders were MA-HPed and their mechanical properties were then compared with the standard (JIS) carbon steels through the well-established heat treatment.

2. EXPERIMENTAL PROCEDURE

Elemental powders of iron (99.5 mass %, 5 μm) and graphite (99.9 mass %, <78 μm) with hypo-eutectoid composition (Fe-0.4 mass % C and Fe-0.6 mass % C) were put into a cylindrical pot (SKD, 450 cm^3) under an argon atmosphere with two types of stainless balls (SUS 304, ϕ 12 mm:ϕ 7 mm = 1:1). The ball-to-powder mass ratio was 8:1. MA was carried out at 120 rpm for 100, 200 and 300 h by using a planetary ball mill (PM400, Retsch Industry Inc.). The MAed powders were characterized by X-ray diffraction analysis (XRD), scanning electron microscopy (SEM) and differential thermal analysis (TG-DTA). TG-DTA was performed under argon atmosphere with heating rate of 0.33 °C/ s. After MA for 200 and 300 h, the Fe/C alloying powders were HPed at various temperatures (below, near and above the eutectoid transformation point A_1) at 41 MPa under nitrogen gas atmosphere. Figure 1 shows various conditions of HP diagrams. The HPed compacts were examined by tensile test at a crosshead speed on 0.5 mm/ min, microstructural observation by using SEM and Vickers hardness measurement.

3. RESULTS AND DISCUSSION

Figure 2 shows SEM images of as-received (a) iron and (b) graphite powders and (c) Fe-0.6C powders mixtures MAed for 300 h. The morphology of iron powders before MA is almost spherical. While, graphite powders have irregular morphology. As shown, after MA for 300 h, the extremely deformed powder particles are observed. The relatively coarsened particles are the aggregates of very fine particles that form due to the repeated forging, work-hardening, fracture and cold-welding during MA. There is no significant change between the morphology of the MAed Fe-0.6C and Fe-0.4C powders mixtures.

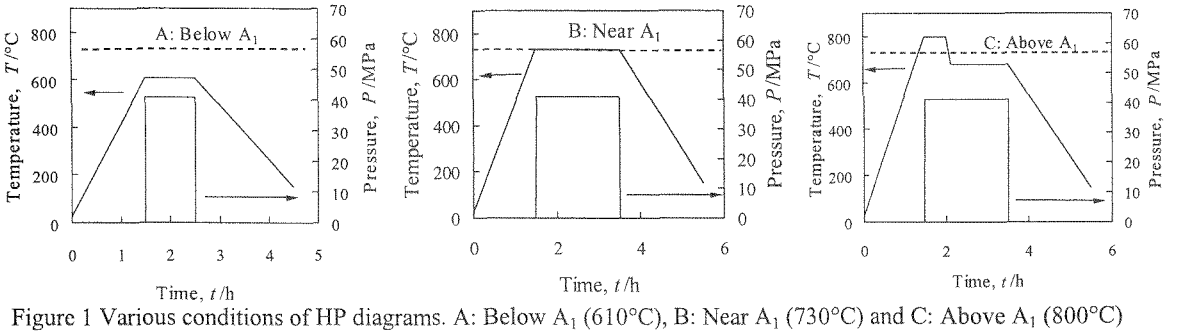

Figure 1 Various conditions of HP diagrams. A: Below A_1 (610°C), B: Near A_1 (730°C) and C: Above A_1 (800°C)

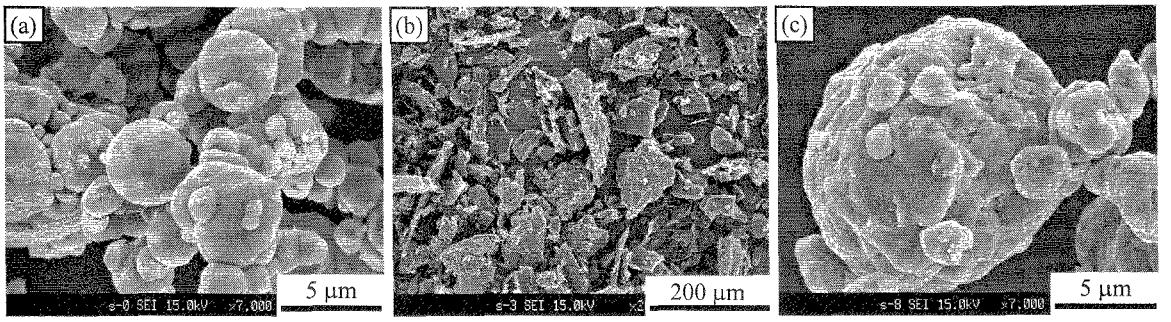

Figure 2 SEM images of as-received (a) iron and (b) graphite powders and (c) Fe-0.6C powders mixtures MAed for 300 h.

Figure 3 shows XRD patterns of Fe-0.6C powders MAed for various time. The XRD peak of graphite does not appear because of the small amount in the mixed powders. The iron peaks broaden and their intensities decrease as the MA time increases up to 200 h. Also their positions shift to the lower angle. However, the intensity of Fe (110) peak increases again when the MA time is 300 h. This indicates that the refinement of crystallite and amorphization take place first[9][13]. Also, many carbon atoms dissolve into the iron lattice to form super-saturated solid solution. When the MA time exceeds 200 h, however, the amorphous Fe/C may begin to transform to another phase such as carbides. Tanaka et al. reported that during MA of Fe-C system, αFe and graphite transformed to Fe_3C through amorphous Fe/C and Fe_7C_3 [9].

Figure 4 shows the changes in the carbon content calculated from the iron lattice parameter using an equation proposed by Fasiska[14] as a function of MA time. The carbon content increases with the increase of MA time up to 200 h and then decreases with further MA time. The highest carbon content (0.064 mass%) obtained corresponds with the broadest iron peaks for 200 h (see Figure 3). The decrease in carbon content shows that the solubility of carbon into the iron lattice has a limit and with further MA time, the super-saturated iron solid solution and amorphous Fe/C tend to transform to another phase, such as carbide Fe_7C_3 or Fe_3C as mentioned previously.

Figure 5 shows DTA-TG curves of Fe-0.4C and Fe-0.6C powders MAed for 200 h. The mass of the MAed powders increases slightly with heating temperature up to 500°C (see the TG curves). This suggests that the oxidation of iron occurs during the heating process due to the impurity of atmosphere gas (Ar). With further heating, the mass decreases because of the oxidation of amorphous carbon to form CO or CO_2 gases. On the other hand, in DTA curves,

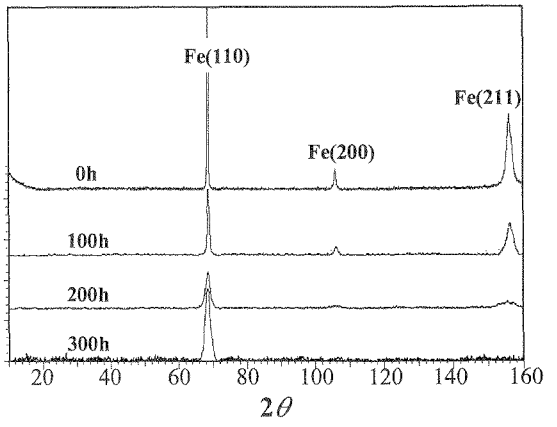

Figure 3 XRD patterns of Fe-0.6C powders MAed for various time.

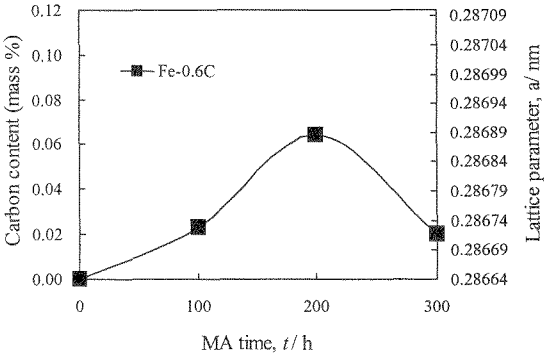

Figure 4 Changes in carbon content and iron lattice parameter as a function of MA time.

exothermic peaks at about 370°C are caused by transformation of super-saturated iron solid solution and Fe/C amorphous phase into ferrite and carbide (Fe_5C_2)

[8-9]. It is confirmed that the intensity for these exothermic peaks decrease significantly for the powders MAed for 300 h because the transformation from the super-saturated iron solid solution and amorphous Fe/C to carbide occurs during MA as mentioned previously. The Fe_5C_2 carbide transforms into the more stable Fe_3C carbide (cementite) with further heating process up to 530°C.

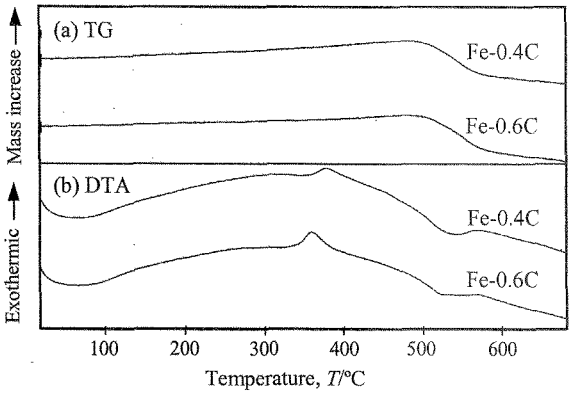

Figure 5 DTA-TG curves of Fe-0.4C and Fe-0.6C powders MAed for 200 h.

Figure 6 shows SEM images of the Fe-0.6C compacts HPed at various temperatures. At low HP temperature (610°C), the compacts compose of nano-size grains (see Figure 6 (a)). However, due to the low temperature, the sintering does not progress well and many voids remain (see Figure 6 (b)). In the case of HP at a temperature near A_1 (730°C) and above A_1 (800°C), both compacts undergo well-sintering process and have almost no voids. Many fine cementite precipitates in the ferrite matrix (Figure 6 (c)). In thermal refining standard steels, such structure is called sorbite. In compacts HPed at 730°C (see Figure 6 (c)), the ferrite grain size is small (about 2.3 μm) showing that the precipitation of fine cementite retards the growth of ferrite grain during the HP process. In the case of HP at 800°C, the sintering progresses well and the coarsening of ferrite grain takes place. Thus the ferrite grain size is large (about 17.4 μm) as shown in Figure 6 (d) and large cementite precipitates at grain boundaries. This structure is similar to that in annealed carbon steel, which is formed by transformation of austenite into ferrite and cementite during the cooling process. There is no significant difference between the microstructures of the Fe-0.4C and Fe-0.6C compacts.

Figure 7 shows the nominal stress-strain curves of tensile test for Fe-0.4C HPed compacts. Where A, B and C are HPed at 610, 730 and 800°C, respectively. No fracture strain is observed in the compact HPed at 610°C (a temperature below A_1), showing that the specimen undergoes a brittle fracture. At a temperature near A_1 (730°C), the strength increases abruptly up to 934 MPa and the fracture strain is about 0.12. However, no yield point is observed. With further temperature increase, at 800°C (above A_1), the fracture strain increases significantly up to 0.28 and the yield point appears clearly. However, the tensile strength decreases rapidly (about 492 MPa).

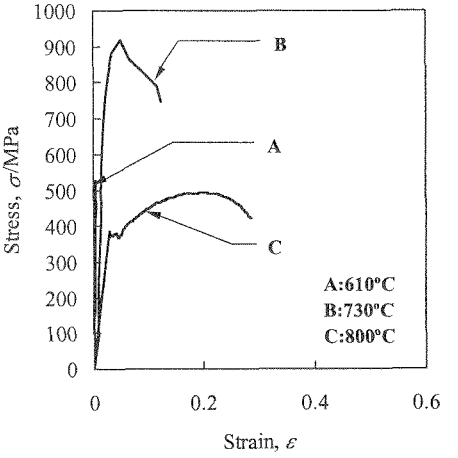

Figure 7 Stress-strain curves of tensile test for Fe-0.4C HPed compacts.

Figure 8 shows the SEM images of the cross section of the fracture surface of the Fe-0.4C compacts HPed at various temperatures after tensile test. In the case of HP at 610°C, the fracture surface consists of many small brittle facets, showing that the sintering process does not progress well because of the low sintering temperature (below A_1). Therefore, the fracture occurs before the yield point and the fracture strain is almost zero. Such brittle fracture is not observed in both specimens HPed at 730 and 800°C. Small and large dimples are observed in the fracture surface of both specimens. However, the dimple size in the specimen HPed at 800°C is larger than that at 730°C, resulting in the increase in fracture strain (see Figure 7). This is because, the enhancement of the sintering process, precipitation of large cementites and ferrite grain coarsening occur during HP. However, the tensile strength is far low compared to that at 730°C. On the other hand, specimen HPed at 730°C has the highest tensile strength (934 MPa) and hardness (257 HV). This is because fine cementites are dispersed into relatively small ferrite grain.

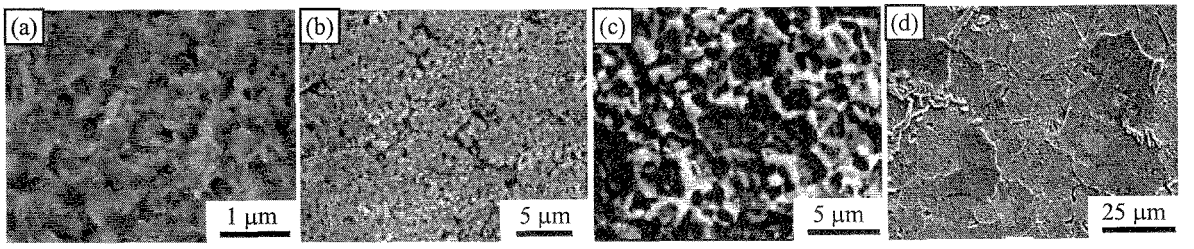

Figure 6 SEM images of the Fe-0.6C compacts HPed at (a) 610°C with high magnification, (b) 610°C with low magnification, (c) 730°C and (d) 800°C.

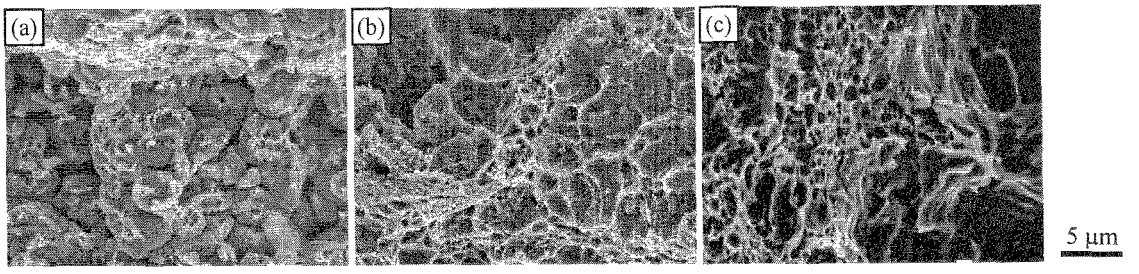

Figure 8 SEM images of the cross section of the fracture surface of the Fe-0.4C compacts HPed at (a) 610, (b) 730 and (c) 800°C after tensile test.

Figures 9 shows the relationship between tensile strength and elongation, while Figure 10 shows the relationship between Vickers hardness and tensile strength of the HPed compacts. A range of data of standard (JIS) carbon steels through normalizing and thermal refining[15] are also given in these figures. The relationships of the compacts HPed at 730°C (near A_1) and 800°C (above A_1) are almost in agreement with those of standard steels. This is because the compacts have microstrctures similar to those of thermal refined steels and normalized steels, respectively. Thus, MA and subsequent HP (consolidation) is a new technique of near net-shape to gain steels with excellent mechanical properties without thermal refining and addition of alloying elements.

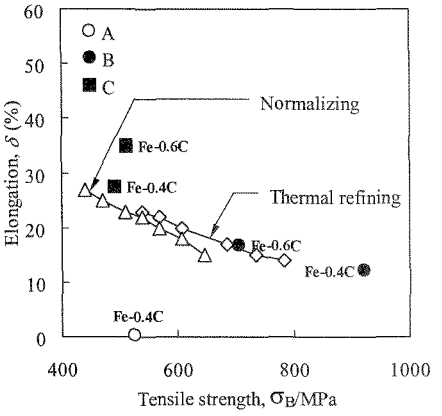

Figure 9 Relationship between tensile strength and elongation of the HPed compacts.

4. CONCLUSION

During MA, refinement of crystallite, formations of super-saturated iron solid solution and Fe/C amorphous phase occur first. With further MA time, these phases begin to transform to more stable phases such as carbides. In the case of HP at 610°C (below A_1), very fine cementites are precipitated in fine ferrite grain with sub-micron meter in the size. However, the good mechanical properties cannot be attained because of low sinterability. At 730°C (near A_1), the strength reaches the maximum value. With further temperature increase (at 800°C), the sintering progresses well and the coarsening occurs, resulting the decrease in the strength. However, the fracture strain increases significantly. The steels obtained in the present study have mechanical properties that are comparable to those of standard (JIS) steels through the well-established heat treatment such as normalizing and thermal refining.

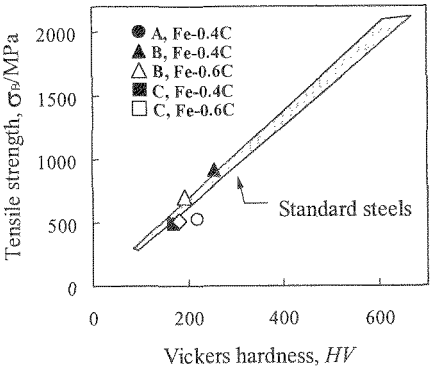

Figure 10 Relationship between tensile strength and Vickers hardness of the HPed compacts

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(Received December 1. 2003; Accepted July 27, 2004)