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# Study on phase transformation of Fe-Ni powders during mechanical alloying

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Fe-Ni ultra-fine particles were prepared by mechanical alloying and phase transformation during mechanical alloying was studied. Results show that phase transformation tendency is different during mechanical alloying of Fe-Ni powders with different nickel content. For Fe-30Ni powder, martensite is the only product obtained with the increase of milling time, while for Fe-35Ni powder, a part of martensite has been transformed into austenite when milling time is prolonged to above 40 h. It indicates that the nickel content plays an important role in the phase transformation tendency during mechanical alloying of Fe-Ni powders. 

## 1. Introduction

In recent years mechanical alloying has gained considerable attention as a nonequilibrium process leading to solid state alloying beyond the equilibrium solubility limit. As a result, a broad range of alloys, intermetallics, ceramics and composites can be produced in the amorphous, nanocrystalline and quasicrystalline state [1]. Besides, among the methods to prepare nanostructured materials, mechanical alloying is promising at a production scale due to its simplicity and low cost.

During mechanical alloying, powder particles are subjected to severe mechanical deformation and are repeatedly deformed, cold welded and fractured, often resulting in solid solution that may be thermodynamically unstable. Previous studies show that both mechanical alloying iron powder with carbon powder or iron-nitride powder and mechanical processing iron powder in a nitrogen gas environment or an anhydrous ammonia atmosphere lead to the formation of nanocrystalline bct-Fe (often referred to as martensite) [2–4]. Martensitic transformation in ultra-fine Fe-Ni particles has been the subject of considerable study since Fe-Ni alloy system is a typical alloy that undergoes martensitic transformation [5–16]. However, so far, limited research has been done on the ultra-fine Fe-Ni particles prepared by mechanical alloying [8, 14–16]. In the present study, phase transformation of Fe-Ni powders with different nickel content during mechanical alloying was studied.

## 2. Experimental

Elemental Fe and Ni (particle size −200 mesh) powders were mixed to get Fe-30wt%Ni, Fe-35wt%Ni. Then the powder mixtures, together with tungsten carbide balls, were sealed in the hardened steel vial under an argon atmosphere. The weight ratio of ball-to-powder was 30 : 1. Mechanical alloying was performed in a QM-I planetary ball mill for different time ranging from 20 h to 80 h. The planetary rotation speed was about 200 rmp. The unmilled and milled powders were characterized by X-ray diffraction (XRD) using Cu $k_\alpha$ radiation. The appearance of particles was observed by means of H-800 transmission electron microscope (TEM). The TEM sample was prepared by dispersing the powder in alcohol using an ultrasonic tank, and transferring a small amount of the solution onto a copper net.

## 3. Results

### 3.1. XRD analysis

Fig. 1 presents the X-ray diffraction patterns of Fe-30Ni and Fe-35Ni powders milled for different time. For the convenience of comparison, the XRD pattern of unmilled powder is also presented, showing that it is composed of $\alpha$-Fe (b.c.c.) and Ni (f.c.c.). The XRD pattern of Fe-30Ni milled for 20 h exhibits a significant broadening of the peaks due to the refinement of grain size and the increase of internal strain. Although it still consists of $\alpha$-Fe and Ni, Ni peak intensity greatly decreases. The lattice parameter of $\alpha$-Fe is about 0.2862±0.0005 nm, which corresponds to the $\alpha'$-martensite phase (b.c.t.). When the milling time is prolonged to above 40 h, martensite is the only product obtained. The microstructural evolution of Fe-35Ni powder is quite different from that of Fe-30Ni during mechanical alloying. The characteristic Ni lines disappear after 20 h of milling. It seems that it takes less time for nickel to dissolve in the iron lattice completely during mechanical alloying of Fe-35Ni. It is quite puzzling and we cannot explain it now. The lattice parameter of $\alpha'$-martensite increases to 0.2864 nm. It is interesting to note that the reverse transformation of martensite into austenite occurs with further milling since the X-ray diffraction patterns of Fe-35Ni powders milled for 40 h and 80 h display the coexistence of the martensite ($\alpha'$) and austenite ($\gamma$). Austenitic volume fraction was estimated in terms of the integrated intensity of the $(220)_\gamma$ and $(211)_{\alpha'}$ peaks. Results show that austenitic volume

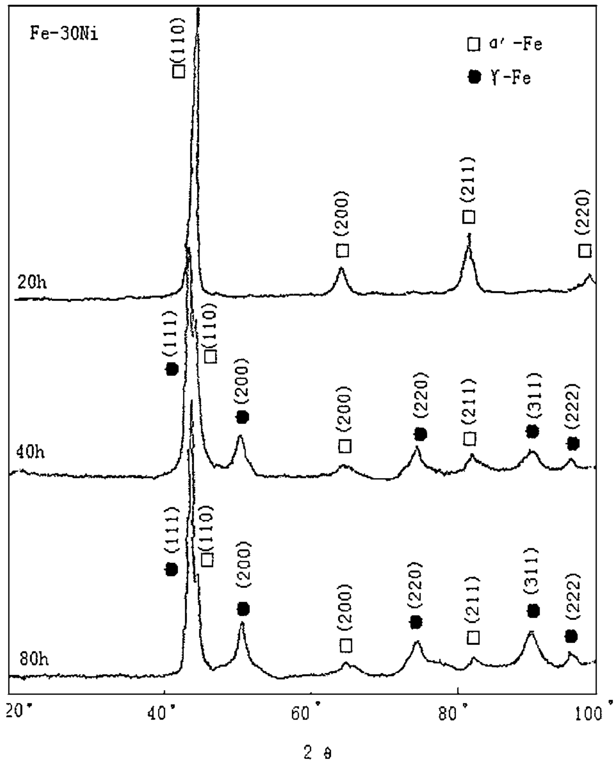



*Figure 1* XRD patterns taken from Fe-Ni powders milled for different time.

fraction increases from 68% for 40 h to 73% for 80 h of milling, indicating austenitic volume fraction increases with increasing milling time.

Single-line approximation was used to extract grain size and microstrain [17], and grain size was evaluated from the breadth of the $\alpha'$ (211) peak. Fig. 2a and b present the grain size and internal strain of Fe-30Ni and Fe-35Ni powders versus milling time. It can be seen that grain size decreases to about 18–20 nm quickly after 20 h of high-energy milling. When milling time is prolonged, the reduction of grain size is accompanied by the increase of the lattice strain level. The lattice parameter of martensite in Fe-30Ni and Fe-35Ni powders is plotted as a function of milling time in Fig. 3. It can be observed that the lattice parameter is in agreement with that of ultra-fine Fe-Ni particles prepared by hydrogen plasma-metal reaction [7, 12] and is almost unchanged with further milling.

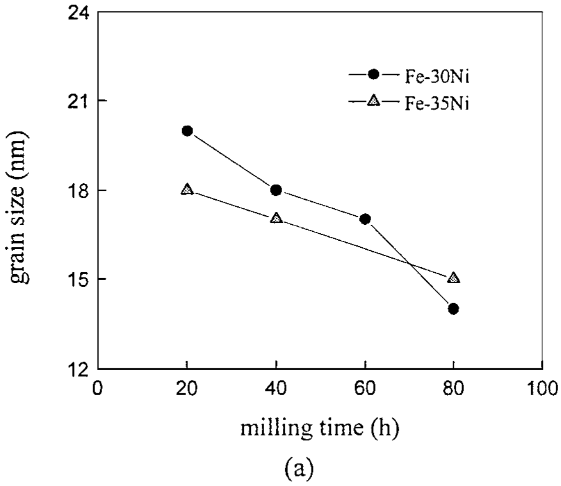



*Figure 2* Grain size (a) and internal strain (b) versus milling time.

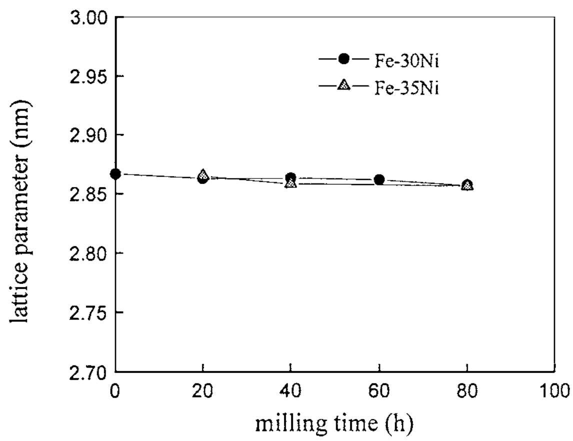



*Figure 3* The lattice parameter of martensite as a function of milling time.

## 3.2. TEM observation

TEM observation on Fe-35Ni powder milled for 80 h shows that the particles do not form clear chains probably due to the existence of austenite and the decrease of magnetic interaction. Fig. 4a is a typical TEM-bright field image of Fe-35Ni powder milled for 80 h. It can be seen that the nm-sized grains tend to agglomerate heavily into larger micron-sized particles. Electron diffraction pattern of the particle is shown in Fig. 4b. It is found that some rings belong to martensite, and the others are identified to be of those of austenite. Therefore, Fe-35Ni powder after 80 h of milling is a mixture of martensite and austenite, which coincides well with the X-ray diffraction analysis.

## 4. Discussion

The schematic of phase transformation during mechanical alloying of Fe-Ni powders is shown in Fig. 5. It can be seen that the martensitic transformation takes place during mechanical alloying of Fe-30Ni, while the reverse transformation of martensite into austenite occurs with further milling of Fe-35Ni powder. Previous reports by several workers show that the austenite becomes stable when the grain size is reduced to nanoscale [5, 6, 9, 13]. From the above experimental results,

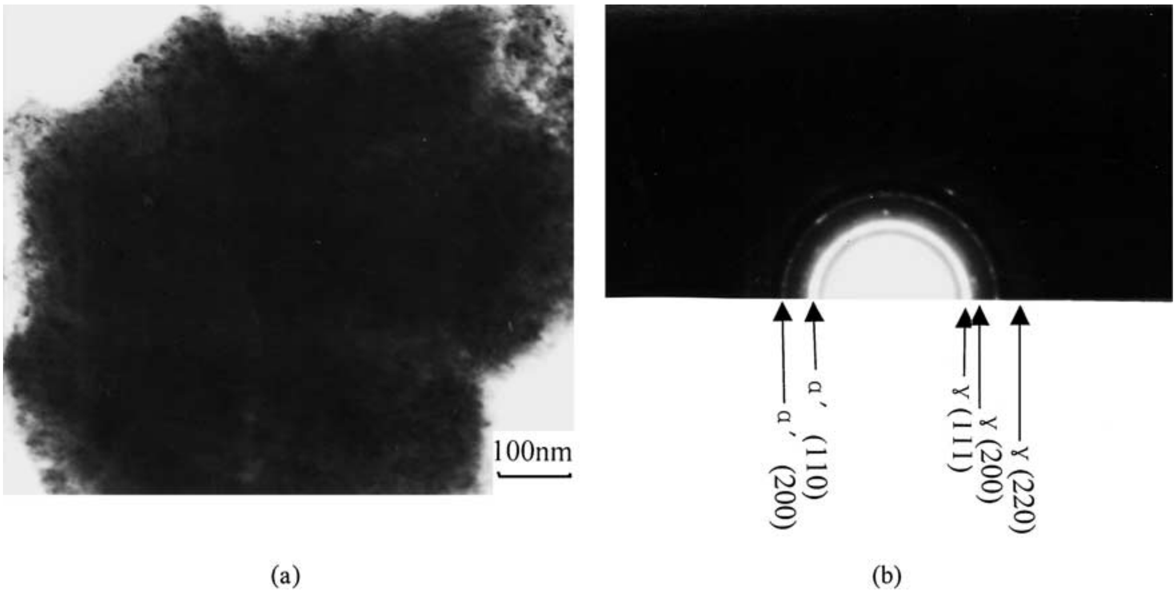



*Figure 4* TEM-bright field image (a) and electron diffraction pattern (b) of Fe-35Ni powder milled for 80 h.

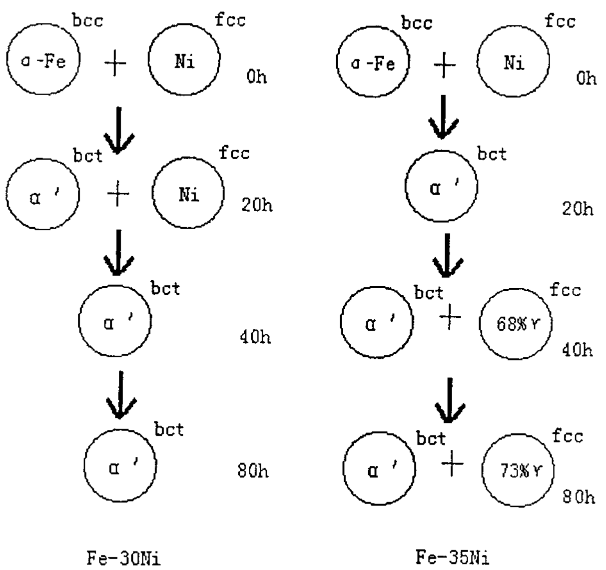



*Figure 5* Schematic of phase transformation during mechanical alloying of Fe-Ni powders.

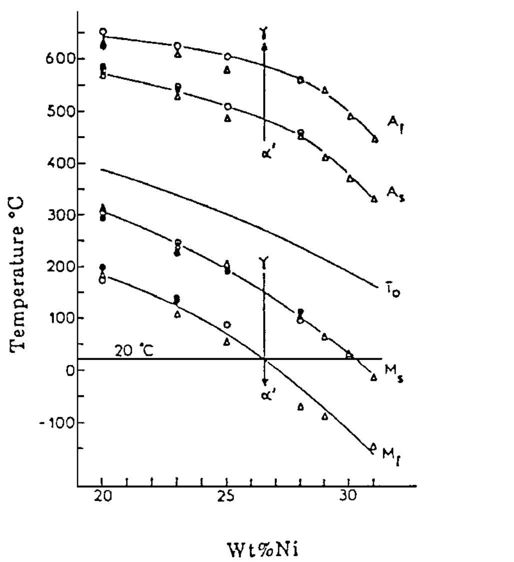


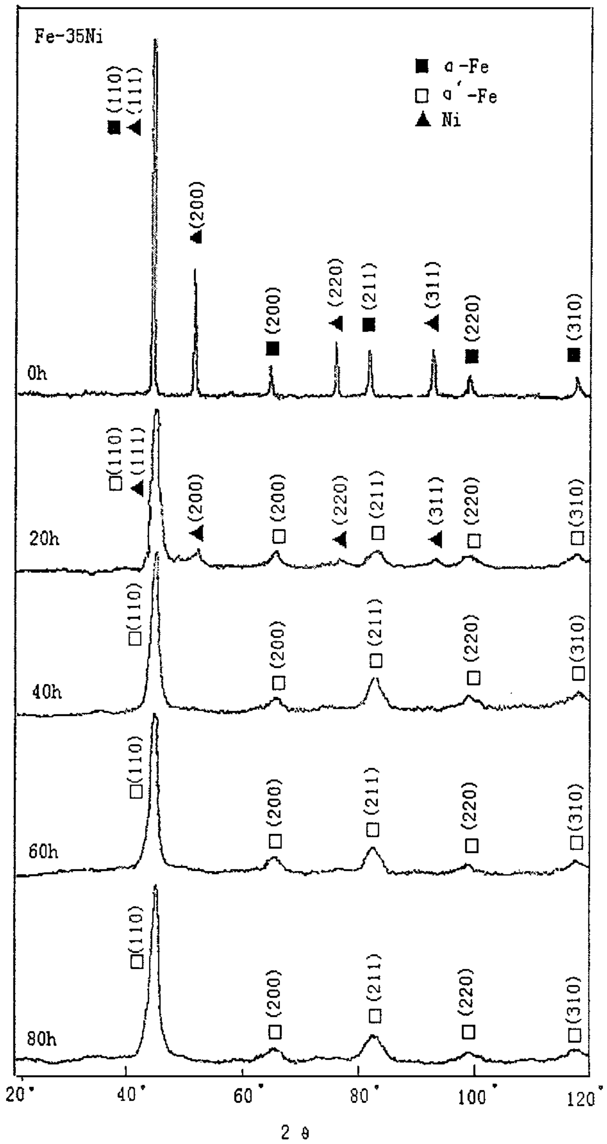

*Figure 6* Composition dependence of transformation temperatures in the Fe-Ni bulk alloys [5].

it can be seen that grain size of Fe-30Ni decreases to 20 nm and Fe-35Ni decreases to 18 nm after 20 h of milling, but in spite of the same milling procedure different microstructures are obtained with further milling. Therefore, the nickel content plays an important role in the phase transformation tendency during mechanical alloying of Fe-Ni powders.

It is well known that mechanical alloying makes it possible to extend the solid solubility limit of Ni in the Fe lattice. With the increase of milling time, more and more nickel atoms dissolve in the iron, accompanied by the reduction in grain size and the increase of internal strain. However, the bct-martensite can't form until the supersolubility level, grain size and internal strain reach a point that the bcc-Fe microstructure becomes unstable. In the present study, for Fe-30Ni, this is a grain size of ≈20 nm, internal strain of ≈0.38%, and nickel concentration of less than 30%. In order to accommodate the presence of extra nickel in the iron lattice and the increase of strain induced by mechanical alloying, a part of energy can be provided by mechanical alloying to transform the lattice from a supersaturated, highly strained bcc-Fe phase into bct-Fe phase with greatly reduced strain.

Referring to the martensite and the reverse transformation temperatures as shown in Fig. 6 [5], the $M_S$ and $M_f$ of the Fe-30wt%Ni bulk alloy are roughly 30°C and −120°C respectively. A mixture of martensite and retained austenite will be obtained at room temperature if cooled quickly. The absence of austenite in Fe-30Ni powder milled for more than 40 h indicates the increase of $M_f$. Though the local material temperature can be raised more than 100 to 150°C above ambient due to mechanical alloying [2], the temperature rise is not high enough to reach the temperature at which transformation of martensite to austenite starts for Fe-30Ni, therefore, the final product of mechanical alloying is martensite.

According to the results of Fe-Ni bulk alloys, the temperatures $M_S$, $M_f$, $A_S$ and $A_f$ decrease with increasing




nickel content. Even though the Fe-Ni bulk alloys with nickel content above 35 wt% are quenched into liquid helium, martensite can not form any longer and thus the bulk alloys remain entirely austenite. Similar phenomenon was found during mechanical alloying of Fe-35Ni powder. When the planetary rotation speed being 230 rmp, it has been confirmed by the XRD analysis that the Fe-35Ni powder milled for 80 h consists of a single phase of the f.c.c. austenite. It means that austenite is more stable than martensite for Fe-35Ni ultra-fine powder, which results in the reverse transformation of martensite to austenite during mechanical alloying. But it is worth noting that austenite in Fe-35Ni powder did not form at the beginning of mechanical alloying, enough energy must be provided to ensure the reverse transformation. It maybe explains why the austenitic volume fraction increases with increasing milling time.

## 5. Conclusions

1. Grain size of Fe-Ni powders decreases to about 18–20 nm quickly after 20 h of high-energy milling. When milling time is prolonged to above 40 h, for Fe-30Ni powder martensite is the only product obtained, while for Fe-35Ni, a part of martensite has been transformed into austenite and austenitic volume fraction increases with the increase of milling time.

2. Phase transformation tendency is different during mechanical alloying of Fe-Ni powders with different nickel content. The martensitic transformation takes place during mechanical alloying of Fe-30Ni, and the reverse transformation of martensite into austenite occurs with further milling of Fe-35Ni. It indicates that the nickel content plays an important role in the phase transformation tendency during mechanical alloying of Fe-Ni powders.

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