A Study about Fe-Ni Mechanical Alloying Process by Dry and Wet Method

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Abstract The evolutions of microstructure of Fe\textsubscript{0.85}Ni\textsubscript{0.15} products, which were prepared by mechanical alloying (MA) with and without process control agent (PCA), were studied using X-ray diffraction and scanning electron microscopy respectively. After MA without PCA (dry method) for 30h, Fe\textsubscript{0.85}Ni\textsubscript{0.15} nanocrystalline alloy powders with bcc-Fe(Ni) phase were obtained; however, powders milled with PCA (wet method) from 20 to 90h, were unalloyed Fe-Ni mixtures with balanced morphology. It seems that dry method works efficiently in nanocrystalline alloying while wet method postpones MA but functionally fines the morphology of materials. Finally, the results were discussed considering the MA kinetics.

Key words mechanical alloying; process control agent; nanocrystalline; morphology

Mechanical alloying has been regarded as one of the most important ways of mixing and combining elements in solid state down to ultrafine scale at moderate temperature since it had been first reported in this application\cite{1}. Process control agent is a medium blended together with the raw materials during milling. Dry method means MA without the use of process control agent (PCA) has been widely chosen in the synthesis of metallic and ceramic ultrafine powders, especially the nanocrystalline alloy powders which were highlighted by experts recently\cite{2,3,4,5,6,7}. Wet method means MA with the use of PCA. A MA method in which mechanochemical reaction was induced has been reported\cite{8}. The effect of diluent in solid state during MA process is shown as decreasing the collision frequency. Ref\cite{9} demonstrates that medium can fine the powders' morphology. However, the affection of medium as PCA, which doesn't participate in any chemical reaction in MA process, has not been specially reported. The most comprehensive way of MA, especially for the synthesis of nanocrystalline alloy, is still under research.

In this paper, products of FeNi-MA using dry or wet method in given hours are presented. By analyzing these products, the character of two methods in MA process will be systematically discussed, considering the MA kinetics. Finally, a constructive conclusion will be given.

1 Experimental

Elemental Fe and Ni powder, both of 99.9% purity or greater, are used as raw materials. The mechanical alloying is performed using QMC1 planetary mill. Each powder mixture in the nominal composition of Fe\textsubscript{0.85}Ni\textsubscript{0.15}, is loaded in a hardened steel container and milled by dry or wet method. As for wet method, anhydrous grain alcohol used as PCA, is filled together with raw materials in the container, with a fit quantity of 20ml/20g powders. The powder/ball ratio is fixed to be 1:20, and mill speeds are 220rpm. Dry and wet samples are obtained in given hours during MA. PCA is removed from the powders before analysis and measurement.

The microstructure of as-milled powders is studied by Philips X'Pert Pro X-ray diffractrometer (XRD) and Hitachi scanning electron microscopy (SEM). Scherrer method is used for calculating the grain size of samples.

2 Results

2.1 Mechanical Alloying Process

Fig.1 shows the x-ray diffraction patterns of Fe\textsubscript{0.85}Ni\textsubscript{0.15} samples milled by dry or wet method for 30h. In the XRD patterns of dry sample, no characteristic diffraction maximums of Ni are visible. Therefore, it is evident that nearly all the Ni atoms are dissolved in Fe crystal lattice. The peaks of α-Fe phase are board due to a grain size of 30nm determined by Scherrer method. This proves that dry sample is nanocrystalline alloy powders with bcc-Fe phase. On the other hand, both the peaks of Fe and Ni are
prominent and in the comparable intensity in the XRD patterns of wet sample, because the powders are unalloyed mixture.

Fig.1 X-ray diffraction patterns of Fe0.85Ni0.15 samples milled by dry or wet method for 30 h

Fig.2 and Fig.3 demonstrate the different evolution of microstructure of Fe0.85Ni0.15 powders in dry and wet MA process respectively. Fig.2 shows that after milled by dry method for 30h, material has the diffraction maximums of Ni located in 52° and 76° disappeared. However, in Fig.3, both Fe and Ni XRD patterns exists and in the comparable intensity no matter how long the sample being milled. All the XRD peaks in two figures broaden as milling continues. It is mainly due to the reduction of grain size. It seems that wet MA contributes little to FeNi alloying while dry method is effective.

Fig.2 X-ray diffraction patterns of Fe0.85Ni0.15 samples milled by dry method for given hours

2.2 Morphology of Particles

The evolution of morphology of the dry milled Fe0.85Ni0.15 particles is shown by the SEM micrographs in Fig.4. We can see that the sample milled by dry method for 20 h is coarse mixture with particle size about 40 µm. In 30 h, particles form the layered structure. Then, as MA continued, particles shatter into smaller fragments, and the particle size reduce.

Fig.3 X-ray diffraction patterns of Fe0.85Ni0.15 samples milled by wet method for given hours

(a) Sample dry milled for 20 h

(b) Sample dry milled for 30 h
The description of MA of ductile alloys which is classically divided into three stages can be supported by Fig.4[9]:

1) Mixing and plastic deformation of particles, as shown in Fig.4a.
2) Welding possesses predominant position with random welding orientation and equiaxed particle formation, as shown in Fig.4b.
3) A steady state processing leads to a rather stable decreasing of particle size, as shown in Fig.4c and d.

Fig.5 shows the SEM micrographs of wet milled Fe0.85Ni0.15 sample after milled for given hours. No layered structure is observed both in 30 h and 90 h during MA. Balanced morphology is characteristic. It’s obvious that wet method is useful to fine and balance the morphology of FeNi particles.

3 Discussion

3.1 Theory of MA
Fracture and welding during shocks in MA are two basic processes, which cause the interdiffusion of matters among material particles such as the dissolving of Ni atoms in Fe crystal lattice in this work. Local stress and local high temperature produced by high-energy milling are the main impetuses of fracture and welding. During collisions, particles are subjected to the increasingly enhanced local inner stress in the process of fracture and welding. At the same time, temperature rises and is sheared by dislocations and nonconservative vacancies generated by itself. These mentioned above contribute to an efficient interdiffusion between elements Fe and Ni. The interdiffusion mixes the two elements down to atomic scale and forming alloy. Temperature rise of powders during MA is believed could be reaching 400 K, content to the thermal activation required for MA.

3.2 Effect of PCA
PCA is an additive in this research, which doesn’t participate in the reaction of raw materials but influence the FeNi MA process indirectly. That is caused by the specific physical effect of this PCA. During MA, alcohol wraps the raw powders and mill balls, forming a buffer area that smoothes the collision intensity. Therefore, the force put upon raw materials
decreases. Simultaneously, liquid alcohol transfers the local high energy produced by collision to the whole system. Therefore the energy of collision is uniformly distributed to the powders. By this way, it is hard to fulfill the requirement of local stress and local high temperature for FeNi alloying, because the high value of these parameters is reduced by the action of distribution-balancing and collision-smoothing.

Therefore, comparing with dry method, wet method provides relatively small stress and temperature to milling powders equally, so shapes balanced morphology of particles, but postpones MA procedure.

4 Conclusions
In this work, we compared the wet method with dry method. Due to the specific effect of PCA on MA, wet method can not provide enough energy for Fe$_{0.85}$Ni$_{0.15}$ alloying. Alloyed powders are not obtained even for sample wet milled for 90h, but the morphology of the sample is fine. By contrast, dry method is effective for the Fe$_{0.85}$Ni$_{0.15}$ alloying which was obtained only after 30h’s dry milling. Therefore, the microstructure and morphology of metal alloys can be conveniently adjusted by dry and wet method respectively. These provide a useful way for the synthesis of material with stricter requirement.

References

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