第15卷 第2,3期
2005年9月

广东有色金属学报
JOURNAL OF GUANGDONG NON-FERROUS METALS

Vol. 15, No. 2,3
Sep 2005

Article ID: 1003－7837(2005)02,03－0255－03

Mechanochemical method for producing iron-based nitrogen-containing nanocrystalline alloys

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Abstract: Iron-based products account the main volume of powder metallurgy production. Nevertheless its strength and reliability are not enough in comparison with classical cast materials. So that is why making nanostructural powder materials allows to increase strength and extend the range of products. A principally new way of nanostructure production is possible by means of iron mechanical alloying with nitride-forming and nitrogen both at the same time. Unlike classical technology of internal nitrogenation, nitrogen saturation, in our case, occurs by whole volume at plastic deformation conditions. A review of experimental results of phase forming alloys in the Fe-Ni, Fe-Ni-Cr, Fe-Ni-N, Fe-Ni-Cr-N, Fe-Cr-Ni systems prepared by mechanical alloying are given. The influence of the technological parameters of mechanical alloying, atmosphere of mechanical activation on nitrogen content and phase composition of examined alloys has been studied. Experimental results of the influence of mechanical alloying technological parameters on degree of ammonia dissociation and nitrogen content in examined alloys are presented. Heat treatment influence of mechanically alloyed, nitrogen-containing alloys on their phase composition and structure are investigated. It was shown that using mechanical alloying, it's possible to prepare high-alloyed iron-based alloys containing more than 1% of nitrogen. It was established that technology of mechanical alloying in ammonia atmosphere allows to prepare austenitic steels with nanocrystalline structure, which affords high value of yield stress. Physico-chemical patterns of interaction between the nitrogen-containing atmosphere and nitride-forming elements under their mutual mechanical activation conditions were established in consequence of theoretical and experimental researches. Some scientific principles of nanocrystalline materials were gained by quantitative description of correlation between the mechanical dose, nitrogen potential, nitrogen content in alloy and morphology of hardening phase.

CLC number: TF 63　　**Document code**: A

At present the use of nitrogen as an alloying γ-phase locking element at both room and low temperatures in Fe-Ni-Cr alloys becomes a very perspective direction in development of new, high-strength non-magnetic materials. The practical application of nitrogen-containing steels will increase because of the necessity of the consumption of efficient alloying elements. Nitrogen as an alloying element is available and has a low cost, and unlike nickel, it doesn't cause allergy in human body. Nitrogen production doesn't need any surface and entrails destruction, which are inevitable in mining. The use of nitrogen allows exchanging nickel and manganese that are the most important elements in alloying of different steels. The technological processes providing the dissolution of nitrogen in iron-based alloys are more complicated than those used with carbon. There are two methods of steel nitrogenation: melt nitrogenation under (normal pressure with

Received date: 2005－08－29

a charge containing nitrided ferroalloys and high nitrogen pressure); nitrogenation of solid alloys (by mechanical alloying in nitrogen containing atmosphere, volume and surface nitrogenation of compact material or powders in the atmosphere of nitrogen-containing gases and hot isostatic pressing (HIP) of powders under nitrogen pressure). Steels containing nitrogen, can be either with equilibrium or with the"superequilibrium"nitrogen contents. "Superequilibrium" nitrogen concentrations are obtained by smelting and crystallization of ingots at high pressure of nitrogen and also by hot pressing of powders under the nitrogen pressure in HIP. In both cases, special and very expensive equipments are required. A principally new way of high-nitrogenous iron alloys production is possible by using mechanical alloying technology[1]. In this case, the introduction of nitrogen occurs in the process of mechanical activation of the starting components in NH_3. The nitrogen content in iron powder can reach 0.6%—0.7% and in composition Fe + 25%Ti more than 1.5%[1,2]. Since nitrogen is a strong austenite-forming element, the study of its influence on phasic composition of mechanically alloyed alloys is very interesting. The purpose of the present paper is to investigate phase-forming in Fe-Ni, Fe-Ni-Cr, Fe-Ni-N, Fe-Ni-Cr-N systems at mechanical alloying.

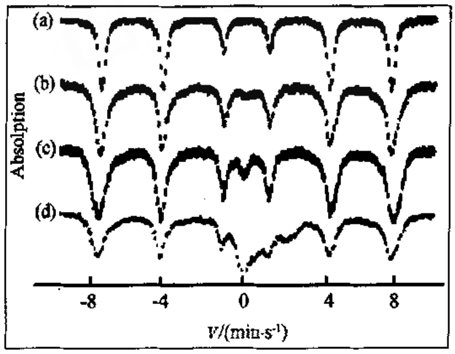

Fig. 1 Mossbauer spectra of $Fe_{78}Ni_{22}$ alloy depending on time of mechanical activation in an NH_3 atmosphere
(a)—40 min; (b)—4 hours; (c)—8 hours; (d)—20 hours

Powders of Fe, Ni and Cr were used as starting components. The mechanical activation of Fe-Ni and Fe-Ni-Cr powders was carried out in the hermetically sealed reactor of an energy-intensive vibrating mill (designed at the Far Eastern State Technical University) in an NH_3 and Ar atmosphere at a vibration amplitude of 90 mm and a vibration frequency from 8Hz to 11 Hz. The mill used as the reactor was filled with steel balls to 0.5—0.6 of its capacity. The ball-to-starting-components mass ratio (intensity ratio) was 20. The time of mechanical activation was varied from several minutes to 20 hours. When the process finished, the final product was investigated by means of γ-ray phase analysis, using a DRON-3 device with CuK_α radiation, and the nitrogen content was determined by a chemical analytical method. Changes in local structure were followed by Mossbauer spectroscopy. The spectra were obtained at room temperature. Then a part of the powder was heated in an argon atmosphere to 700 ℃ and held 1 hour.

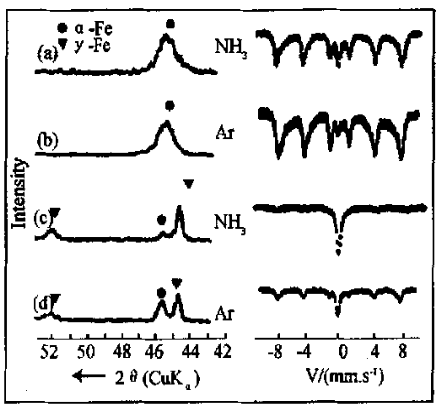

Fig. 2 Mossbauer spectra and diffractograms of $Fe_{72}Cr_{18}Ni_{10}$ alloy depending on mechanical activation atmosphere
(a), (b) alloys after 8 hours of mechanical activation ; (c), (d) alloys after the annealing at 700 ℃ for 1hour

Figs. 1 and 2 show Mossbauer spectrums and difractograms of $Fe_{72}Cr_{18}Ni_{10}$ and $Fe_{78}Ni_{22}$ alloys depending on time and atmosphere of mechanical activation. According to given experimental results, the main influence on formation of γ-phase among examined technological parameters was made by vibration frequency of mechanoreactor (Figs. 1 and 3). This dependence is clearly displayed in case of mechanical activation of examined compositions in an NH_3 atmosphere (Fig. 3).

Mechanical activation in an NH_3 atmosphere leads to an increase in content of γ-phase in examined systems. For example, mechanical activation of $Fe_{72}Cr_{18}Ni_{10}$ in an NH_3 atmosphere for 8 hours allows to extract about 90% of austenite after the annealing at 700℃ (Fig. 2). It was established, that mechanical alloying technology in ammonia atmosphere allows to prepare austenic steels with nanocristalic structure, which affords high value of yield stress.

Thus, mechanical alloying of iron by austenite-forming elements allows to gain γ-phase. Maximum quantity of γ-phase, both in two components Fe-Ni system and in poly-components Fe-Ni-Cr system is gained by this mechanical alloying in an NH_3 atmosphere. The method of mechanical alloying can be successfully used in iron-based alloys with high content of nitrogen.

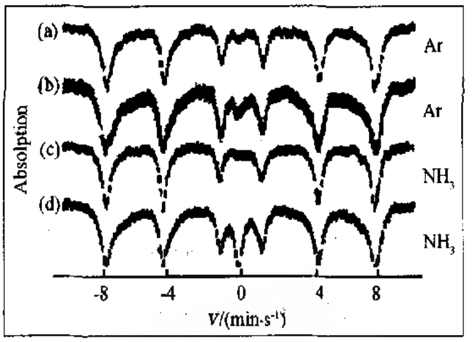

Fig. 3 Mossbauer spectrums of $Fe_{72}Cr_{18}Ni_{10}$ alloy in an NH_3 and Ar atmosphere depending on vibration frequency of mechanoreactor and mechanical activation atmosphere

(a) −8 Hz; (b) −12 Hz; (c) −8 Hz; (d) −12 Hz

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