



INVESTIGATION OF THERMAL STABILITY OF NANOCRYSTALLINE STRUCTURE HIGH-NITROGEN AUSTENITIC POWDER STEEL OBTAINED BY MECHANICAL ALLOYING

Anatoly A. Popovich, Nikolay G. Razumov and Evgeniy L. Gyulikhhandanov
Peter the Great Saint-Petersburg Polytechnic University, Polytehnicheskaya 29, St. Petersburg, Russia
E-Mail: popovicha@mail.ru

ABSTRACT

Mechanical alloying (MA) is a solid-state powder technique involving repeated welding, fracturing, and rewelding of powder particles in a high-energy ball mill. MA has now been shown to be capable of synthesizing a variety of equilibrium and non-equilibrium alloy phases starting from blended elemental or prealloyed powders. The non-equilibrium phases synthesized include supersaturated solid solutions, metastable crystalline and quasicrystalline phases, nanostructures, and amorphous alloys. Currently, there is a question of stability of nanocrystalline structure obtained by mechanical alloying.

Keywords: high-nitrogen austenitic steel, powder alloys, mechanical alloying, phase formation, nanocrystalline structure.

INTRODUCTION

Mechanical alloying (MA) is a powder processing technique that allows production of homogeneous materials starting from blended elemental powder mixtures. MA is described as a high energy milling process in which powder particles are subjected to repeated cold welding, fracturing, and rewelding [1]. MA is carried out at a comparatively low temperature, where the formation of an ideal crystal structure is difficult. This opens a route to the synthesis of substances and materials in nanocrystalline and amorphous states [2, 3]. The advantage of using MA for the synthesis of nanocrystalline materials lies in its ability to produce bulk quantities of material in the solid state using simple equipment and at room temperature. The first report of formation of a nanostructured material synthesized by MA is by Thompson and Politis in [4]. In [5] Koch has summarized the results of the synthesis and structure of nanocrystalline structures produced by mechanical attrition.

Nanocrystalline structures, produced by MA are characterized by the presence of high densities of lattice and grain boundary dislocations that create long-range fields of elastic stresses. As a result, there are significant atomic displacements from the nodes of the ideal crystal lattice, therefore, obtained via MA nanostructures have a high stored energy and are metastable. This leads to rapid development of recovery processes and grain growth at high temperatures [6, 7]. This significantly limits the potential of using them. In this regard, the question of the thermal stability of nanocrystalline structures formed in metals and alloys by severe plastic deformation represents great scientific and practical interest. The purpose of the present work is to investigate stability of nanocrystalline structure high-nitrogen austenitic powder steel obtained by mechanical alloying.

EXPERIMENTAL

Powder of high-nitrogen austenitic stainless steel was prepared by mechanical alloying (MA) as described in

[8, 9]. The heating is maintained in a horizontal through passage furnace OTF-1200X-HP-55 (MTI Corp.) in argon atmosphere. Changes in the phase composition and lattice parameters of the materials were determined by X-ray analysis using a Bruker D8 Advancer with $\text{CuK}\alpha$ -radiation (voltage - 30 kV, current - 30 mA). Microstructure analysis was carried out using an optical microscope Leica-DMI 5000 at magnifications of x50-x1000. Fine structure of the powder was studied by high-resolution transmission electron microscope JEOL-2100F with an acceleration voltage of 200 keV. Obtained powders were compaction by hot rolling and sparks plasma sintering (SPS). Powder rolled in the shell at $T = 900^\circ\text{C}$. SPS process was carried out at $T = 900^\circ\text{C}$ and pressure 49 MPa using FCT HP D 25.

RESULTS AND DISCUSSIONS

Investigating the influence of MA parameters on the phase composition of the alloy showed that the first of the alloying elements dissolved in the lattice of iron is nickel, then chrome and manganese (Figure-1). The reason for this regularity is that the alloying elements Ni, Cr, Mn form a substitution solid solutions with iron, and nickel has the closest to Fe atomic radius - 126 pm, followed by chromium and manganese. It is found that with the increasing time of mechanical alloying the amount of undissolved alloying elements gradually decreases and, at the same time, the process of restructuring the bcc lattice in fcc begins. After 30 min of MA the contents of γ -phase in the alloy reaches $\sim 3 - 5\%$ (vol.). It is obvious that the rearrangement of the crystal lattice occurs without heating, and the formed γ -phase has a nanometric dimensions. With further increase of the MA duration alloying elements are completely dissolved in the iron and, accordingly, the share of γ -phase in the structure increases while the share of α -iron - decreases.

Severe plastic deformation leads to the formation of a uniform nanocrystalline structure in high-nitrogen austenitic stainless steel (Fe-18Cr-8Ni-12Mn-N system) even at room temperature (Figure-2a). Nanocrystal sizes



are in the range from 6 to 20 nm, there are a lot of dislocations on grain boundaries and atomic planes are partially coherent. However, there are a lot of boundaries whose images are poorly defined and diffraction contrast in the grains is heterogeneous and often changes in a complicated way, which indicates a high level of internal stresses and elastic distortion of the crystal lattice. This complicated contrast is present both in the grains, containing lattice dislocation, and in the defect-free grains, indicating that sources of internal stresses are grain boundaries. A detailed study of structure at higher magnification, when it is possible to resolve the individual atoms of the crystal lattice, allowed concluding that the grain boundaries are periodically staggered formations. The images of atomic planes near the grain boundaries often show a significant distortion or bending of the crystal lattice. Some images of the atomic planes are cut off, which indicates the presence of dislocations. The diffraction pattern obtained from the area of 0.5 microns is a set of reflexes, which are located on concentric circles (Figure-2b). This is evidence of large-angle misorientations of neighboring grains in highly deformed structure and considerable internal stresses. Reflexes on the circles are associated with diffraction by the crystal lattice of iron.

Structural properties of nanocrystal grains, investigated by the high-resolution electron microscopy, are quite similar and comply with the computed atomic structure of two-dimensional nanocrystalline materials [10], where the majority of grain boundaries are large-angular arbitrary boundaries. These data are in line with the diffraction studies. In addition, grain boundaries are usually narrow: their width is about 1-5 interatomic

distances, which is close to the width of the grain boundaries in conventional coarse materials.

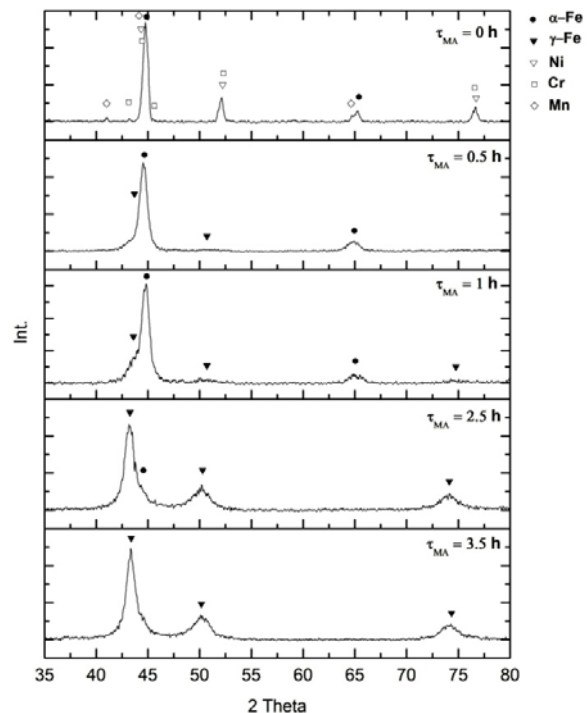


Figure-1. XRD patterns of the powder Fe-18Cr-8Ni-12Mn-xN for different MA durations.

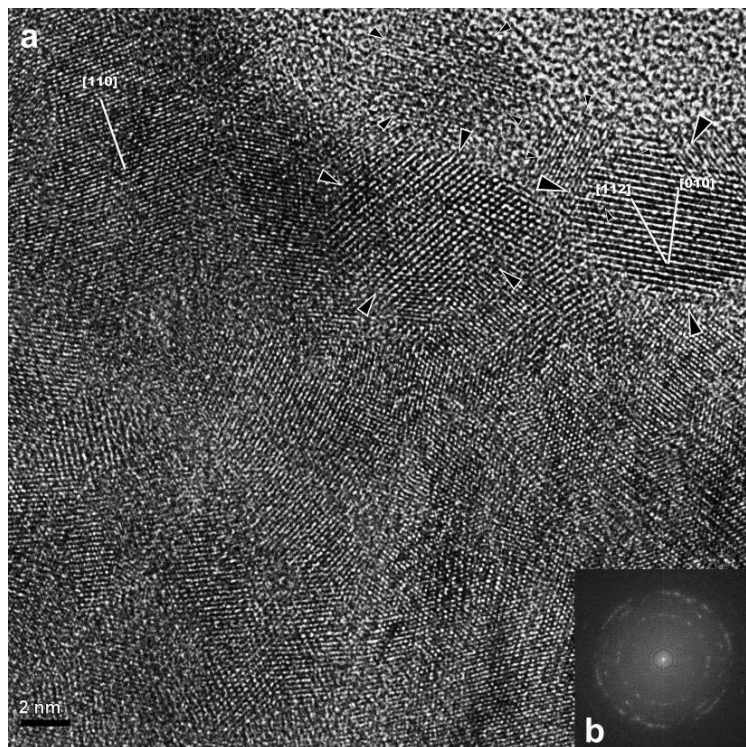


Figure-2. Nanocrystalline structure of high-nitrogen powder alloy Fe-18Cr-8Ni-12Mn-0.9N, after MA for 3.5 hours in the atmosphere of ammonia (a) and electron diffraction pattern (b).

The resulting structure was very stable that confirmed by the complete resistance to chemical etching. Obtained powder had high hardness (650 HV) and strength with the substantial absence of ductility. However, high temperature (1150 - 1200 °C) and long

enough (1 - 1.5 hours) heating have led to a typical etched pattern of recrystallization firstly with fine grain 1-2 μm (Figure-3a), and further to large grain - up 6 μm (Figure-3b). Recrystallization leads to a reduction hardness to 420-450 HV.

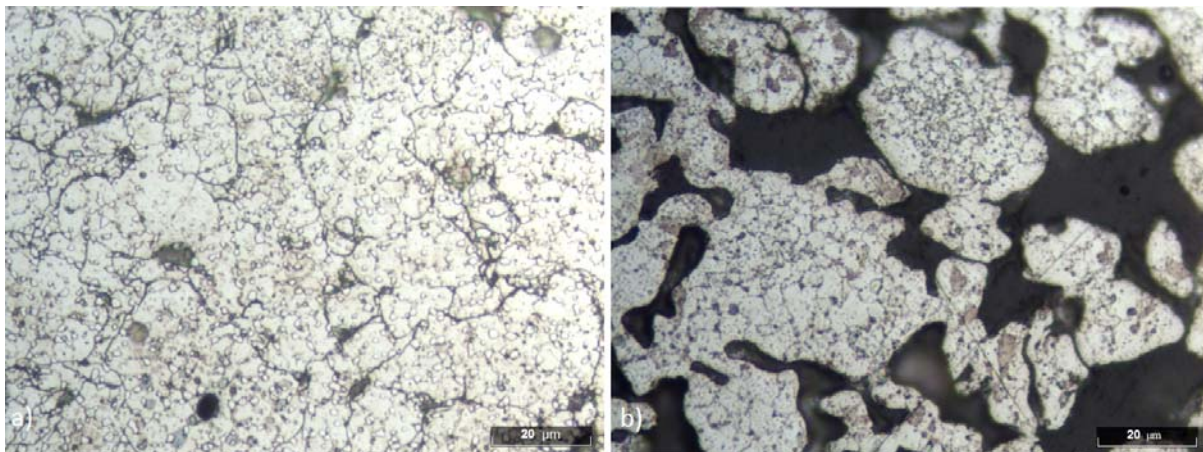


Figure-3. Microstructure of high-nitrogen powder alloy Fe-18Cr-8Ni-12Mn-0.9N after prolonged high temperature heating: a) 1180 °C, 1 hour; b) 1180 °C, 1.5 hours.

In [11], it was revealed in the diffusion nitrided alloyed ferrite that a nitriding zone has been recrystallized with the formation of new grains. Since there are no nitrides in this zone, or mixed segregations of nitrogen atoms and an alloying element, which have a high affinity

to nitrogen (type of Guinier-Preston zones), it was suggested that this zone causes recrystallization. Subsequently, these phenomena have been studied and identified as a complex reaction of recrystallization and emissions.



Experiments of the deformation effect showed the fundamental possibility of obtaining a compact material from the powder of high-nitrogen austenitic steel obtained

by mechanical alloying, with a tensile strength of more than 1100 MPa (Table-1).

Table-1. Mechanical properties of high-nitrogen powder alloy Fe-18Cr-8Ni-12Mn-0.9N compaction by hot rolling and SPS.

Method of compacting	N, %	R _{p0.2} , MPa	R _m , MPa	δ, %	HV
Hot rolling at T=900°C in 2 steps with reduction 50% + quenching at T=1150°C	0,75	850	970	15,0	470
SPS + Hot rolling at T=900°C in 2 steps with reduction 50% + quenching at T=1150°C	0,70	1030	1170	16,0	540

Thus, to create constructional steels and alloys with necessary ductility, high stability of nanocrystalline state may be an obstacle in a process chain of product manufacture, and only controlled degradation of such structure may lead to desired results. The whole question boils down to: is it possible to restore such a state again after the destruction.

CONCLUSIONS

It has been shown that severe plastic deformation leads to the formation of uniform nanocrystalline structure in high-nitrogen austenitic stainless steel even at room temperature. Nanocrystalline structure of these alloys consists of two structural components: grains-crystallites and grain boundary regions, what corresponds to computed atomic structure of nanocrystalline materials. Dimensions of nanocrystals are from 6 to 20 nm. To create constructional steels and alloys with necessary ductility, high stability of nanocrystalline state may be an obstacle in a processing chain of product manufacture, and only controlled degradation of such structure may lead to desired results.

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