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FORMATION OF A FINE-GRAINED STRUCTURE OF IRON-NICKEL ALLOYS BY REVERSIBLE MARTENSITE TRANSFORMATION

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We have investigated the influence of multiple γ–α–γ transformations proceeding by the shear mechanism on the structural-phase state of austenite in iron-nickel alloys N28T2Yu2 and N32. It has been shown that it is possible to form, by cyclic γ–α–γ transformations, a fine-grained structure of austenite due to the fragmentation of the initial structure that develops under the conditions of progressing disorientation of the crystal lattice of the γ-phase. In the process of thermocycling, the reversed austenite was continuously strengthened under the condition of the development of disorientation of its lattice.

Introduction. The physicomechanical properties of metal alloys can be improved by refining the structure to the ultradisperse level. Unlike the current methods of obtaining fine-grained materials that are mainly power-intensive (methods of powder metallurgy, intensive plastic deformation, spinning, crystallization of amorphous materials, etc.), for iron-based metals a method of refining the structure by means of cyclic direct γ–α and reverse α–γ transformations can be developed. This method is a further development of the method of hardening metastable austenite alloys by cyclic martensite transformations (phase cold working method) which was first proposed at the Institute of Physics of Metals, Ural branch of the Russian Academy of Sciences [1].

The mechanisms of formation of the structural-phase state of metastable iron-based alloys in the process of cyclic martensite transformations are determined by the ability of the structure to accumulate structure defects and internal stresses that arise from such transformations, i.e., by the degree of phase cold hardening [1–4]. It has been shown experimentally that the reproducibility of the initial austenite structure of iron-based alloys as a result of cyclic γ–ε–γ and γ–ε′–γ transformations accompanied by a restructuring of the crystal lattice fcc–fccp–fcc and fcc–18R–fcc respectively turned out to be much higher than iron-nickel-based alloys in which γ–α–γ transformations with an fcc–bcc(bct)–fcc restructuring occur [15]. Such a mechanism was explained by the fact that as a result of cyclic γ–α–γ transformation (volume effect up to 3–4%), the dislocation density increased by more than two orders of magnitude, whereas as a result of γ–ε–γ transformations (volume effect 1.75%) it increased by no more than one order of magnitude, and as a result of γ–ε′–γ transformations (volume effect 0.5%), it remained practically unaltered.

Under the conditions of generation of dislocations and their accumulation and interaction additional subboundaries can be formed, e.g., through the formation of polygonal walls by dislocations of one sign. Due to this a fragmented structure of reversed austenite (that has passed a cycle of γ–α–γ transformations) arises. The packing defects that appear in iron-manganese alloys as a result of cyclic martensite transformations did not lead to the formation of additional subboundaries and fragmented structural components. Therefore, of the martensite transformations of different types in iron-based alloys, only γ–α–γ transformations having a minimum volume effect are able to effectively refine the structure of austenite.

Below we present the results of investigating the influence of multiple γ–α–γ transformations in iron-nickel alloys N32 and N28T2Yu2 on the development of fragmentation of the crystal structure of reversed austenite and the possibility of formation of fine-grained structures by this mechanism under the conditions of accumulation of defects in the crystal structure.

X-ray structural studies were carried out on monocrystalline specimens in an RKV-86 rotating-crystal X-ray camera. To this end, from coarse-crystalline ingots obtained by slow cooling of the melt in a thermal high-frequency

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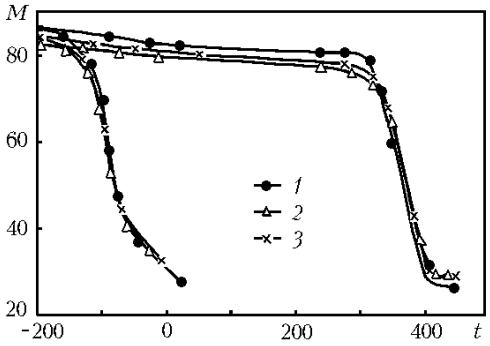


Fig. 1. Magnetometric curves of the cooling and subsequent heating of the N32 alloy in the initial state (1) and upon the first (2) and hundredth (3) γ–α–γ transformations. *M*, %; *t*, °C.

furnace, cylindrical monocrystalline specimens of diameter (0.8–1.0) mm with a crystallographic orientation of the specimen axis close to the direction $[100]_\gamma$ were cut. For magnetometric studies, we used polycrystalline specimens of diameter 8 mm and length 20 mm obtained from a coarse-crystalline ingot by forging. The deformed layer of all specimens was etched chemically.

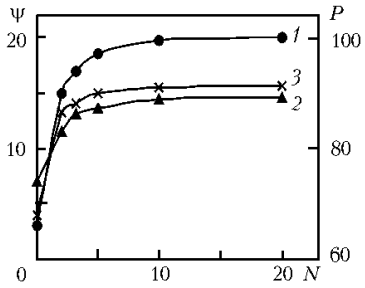
The use of monocrystalline specimens for X-raying has made it possible to observe the disorientation of the austenite lattice from fractions of a degree to several tens of degrees and thus follow the development of the process of fragmentation and refining of the structure. The necessary X-ray reflections of austenite and martensite were obtained by rocking the specimen in the interval of angels of the X-ray chamber limb determined from the preliminarily constructed pole figure of the initial austenite with account for the orientation relations between the austenite and martensite lattices. For the calculations we assumed that the orientation relations corresponded to the Kurdyumov–Zaks condition. The maximum angle of lattice disorientation ψ characterizing the degree of fragmentation of the structure was calculated from the azimuthal diffusion of the austenite $(200)_\gamma$ and martensite $(002)_\alpha$ reflections which were situated near the equator of the X-ray photograph.

The microstructure was investigated by means of an "Epiquant" optical microscope. Electron-microscope studies were made on a PRÉM-200 microscope. The hardness was measured on plates cut from the same coarse grains from which X-ray specimens were cut. The temperature points of the beginning and end of direct and reverse transformations were measured by the differential magnetometric method. The thermomagnetic curves were obtained in the temperature range including the γ–α and α–γ transitions.

Cooling of the alloys in liquid nitrogen led to the formation of 80–85% of the martensite phase. Heating of hardened specimens to the temperature of the end of the reverse α–γ transformation decreased the completeness of the direct martensite transformation that followed by 3–8%.

Multiple cycles of γ–α–γ transformations at a heating rate of 60–80 deg/sec in the range of the α–γ transformation caused an additional stabilization of austenite with respect to the subsequent direct martensite transformation. Because of the insignificant stabilizing action of γ–α–γ transformations on austenite and the ability of iron-nickel alloys for multiple martensite transformations (Fig. 1), it was possible to investigate the process of development of fragmentation of the austenite and martensite structure depending on the number of γ–α–γ transformations and the degree of phase cold working.

The X-ray photographs of monocrystalline specimens upon reverse α–γ transformation have shown that all reflections of austenite diffused in the azimuthal direction. On the pole figure the reflection centers of the initial and the reversed austenite coincided within the measurement accuracy (one-two degrees). No additional austenite reflections were observed. This points to the absence of multiplication of orientations of the reversed austenite in accordance with the orientation relations and to the realization of the mechanism of structure refinement only due to the development of its lattice fragmentation. With increasing number of transformation cycles the azimuthal diffusion of reflections monotonically increased and the Debye rings in the X-ray photograph were filled in the azimuthal direction. The angle ψ increased therewith (Fig. 2).



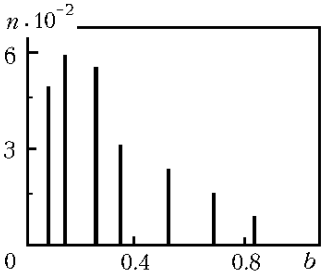


Fig. 2. Change in the maximal disorientation angle ψ of the crystal lattices of austenite (1), martensite (2), and austenite hardness *P* (3) depending on the number of γ–α–γ transformations in the N32 alloy. ψ, deg; *P*, HRB.

Fig. 3. Histogram of the fragment sizes of the reversed austenite of the N28T2Yu2 alloy upon 80 γ–α–γ transformations. *b*, μm.

The X-ray photograph of the rotation of monocrystalline specimens of the investigated alloys upon 35–50 γ–α–γ transformations had the form of the diffraction pattern of a grain-oriented polycrystal. The martensite reflections therewith turned out to be diffused. Upon 80–120 γ–α–γ cycles the diffraction pattern contained practically solid lines of austenite. This was an indication of complete phase recrystallization of austenite and transformation of the initial monocrystal to a polycrystalline specimen. This process proceeded in the investigated alloys qualitatively in the same way, but in the N28T2Yu2 alloy it developed more intensively. The X-ray photographs of a stationary specimen showed some variation in the intensity of the diffraction lines in the azimuthal direction, which was somewhat smaller for the martensite lines. The different azimuthal diffusion of reflections of the γ and α phases determined the different degree of fragmentation of the crystal lattices of austenite and martensite. This was due to the fact that the mechanisms of accumulation of disorientations of the fcc and bcc lattices can differ [6]. Measurements of the hardness *P* of the reversed austenite have shown that it increased upon the first γ–α–γ cycle by 32% compared to the initial austenite. Subsequent thermocycling led to a monotone increase in *P*. In so doing, the degree of hardening of austenite of the N32 alloy correlated with the development of disorientation of the lattice of the initial phase (Fig. 2, curve 3). Similar dependences were also obtained for the N28T2Yu2 alloy.

Electron-microscopic studies have shown that in the process of thermocycling in the reversed austenite there appeared additional subboundaries formed by the dislocations generated by the γ–α and α–γ transformations. At a certain stage the subboundaries formed pronounced fragments in the initial austenite grains. Concurrently with the development of azimuthal diffusion of reflections in the electron diffraction patterns, upon 10–20 cycles subsequent decomposition of reflections into 3–5 components was observed, which pointed to the formation at this stage of additional subgrain boundaries. The size of fragments in the reversed austenite decreased with increasing number of transformation cycles. Upon 30 cycles a large number of fragments of size 0.2–0.8 μm were observed. As a result of 80–100 cycles the size of fragments reached a nano-scale level of 0.08–0.1 μm for the N28T2Yu2 alloy and 0.1–0.15 μm for the N32 alloy (Fig. 3). Multiple thermocycling (over 30 cycles) caused the formation of twins of the reversed austenite. The volume fraction of twins increased with increasing number of γ–α–γ transformations as a result of the accumulation of internal stresses in the γ-phase.

Thus, multiple thermocycling of the N32 and N28T2Yu2 alloys with the participation of direct γ–α and reverse α–γ martensite transformations led to a fragmentation of the initial austenite to a nano-scale level (nano-fragmentation). A decrease in the heating rate in the range of reverse transformation to 3–6 deg/sec led to a decrease in the disorientation angle ψ due to the occurrence of relaxation processes in the martensite-hardened alloy, as well as to the enrichment of retained austenite with nickel at the cost of the corresponding depletion of martensite. Repeated cycles led to a decrease in the temperature of the beginning of direct transformations and in the quantity of cooling martensite. Under such conditions it was no longer possible to attain recrystallization of the initial austenite by means of fragmentation of its lattice.

The recrystallization of the austenite of iron-nickel alloys as a result of multiple γ–α–γ cycles can be represented as the formation of additional subboundaries by the interacting dislocations under the conditions of accumula-

tion of dislocations. In so doing, it is important that defects in the crystal structure that arise in the reversed austenite are more thermostable than the defects formed in the process of plastic deformation [7]. As a result of this, they can remain at reverse transformation temperatures and accumulate in the subsequent thermocycles. Part of the dislocations generated by both γ–α and α–γ transitions gather at the subboundaries, and this determines their angular disorientation and the sizes of new subgrains.

It should be noted that the fragmented structure formed by multiple γ–α–γ martensite-type transformations differs from the normal polycrystalline structure mainly in that the polycrystalline aggregate grains are bounded by large-angle boundaries and the fragments or subgrains are bounded by small-angle ones. This fact may turn out to be essential for the formation of the complex of physicomechanical properties, since it is known that small-angle boundaries with disorientation angles up to 10^{o} have a higher mechanical stability. The fragmented structure of the phase-cold-worked austenite and martensite can appreciably speed up the diffusion processes, especially on the subgrain boundaries, and, as a result of this, intensify the saturation with doping elements because upon γ–α–γ transformations a large extent of subgrain boundaries and a high density of dislocations on both the subboundaries and in the bulk of fragments are observed.

Conclusions. As a result of cyclic γ–α–γ transformations with a diffusionless character of the reverse α–γ transition in the iron-nickel alloys N32 and N28T2Yu2 a submicroscopic or a nanocrystalline structure of the reversed austenite was formed due to the progressing fragmentation of the lattice of the initial γ-phase. Already at the initial stage of thermocycling (10–20 cycles) additional closed subboundaries were observed.

As a result of 80–100 cycles, the size of fragments reached a nano-scale level. Upon multiple thermocycling (over 50 cycles), twins of the reversed austenite were formed and their volume fraction increased with increasing number of γ–α–γ transformations. The mechanism of phase recrystallization of austenite was realized without involving recrystallization processes. The degree of hardening (hardness) correlated with the development of disorientation of the initial-phase lattice.

The realization of the method of cyclic martensite transformations requires only thermal cycles of cooling and heating in a technologically convenient temperature range. There is no need to perform complex operations associated with the melting and evaporation of the materials, the intensive plastic deformations, etc. The formation of the highly disperse state of the alloys can be used to improve the physicomechanical properties of metastable iron-nickel alloys. The fields of practical application of alloys with highly disperse austenite and martensite are different. Single-phase austenite alloys find application as high-strength nonmagnetic materials. Two-phase alloys with highly disperse martensite and retained austenite are of interest for creating high-strength materials due to the fact that nanocrystalline slightly magnetic austenite upon a partial α–γ transition is able to break the domain structure of ferromagnetic martensite and increase the coercive force of high-strength martensite-aging steel [8].

NOTATION

b, size of fragments of the crystal lattice of austenite, μm; *M*, volume fraction of the martensite phase, %; *n*, number of fragments of a given size; *N*, number of γ–α–γ transformations; *P*, hardness, HRB; *t*, temperature, ^{o}C; Ψ, disorientation angle of the crystal lattice, deg.

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