GRAIN STRUCTURE OF Ni$_3$Fe ALLOY

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The grain structure of a Ni$_3$Fe polycrystal is studied by means of optical metallography taking into account the type of grain boundaries. Grains with random boundaries having special boundaries on the inside are revealed. Grains are classified by the presence and position of special boundaries. The energy of different types of grain boundaries is estimated.

The mechanical properties of alloys are largely dependent on the grain structure. The latter is determined by the fundamental Hall–Petch relation whose applicability has been proved by a large body of present-day research [1]. In addition to the quantitative characteristic of the grain structure – the average grain size – the types of grain boundaries influence mechanical properties. In [2–4], for instance, the deformation stress is shown to depend on the presence of special boundaries in the materials of interest. The types of grain boundaries are also responsible for the development of high-temperature creep and grain-boundary cracking, segregation distribution over grain boundaries, etc. In this connection, it is important to describe materials according to the types of grain boundaries. There are a number of approaches to the grain-boundary classification: by grain-boundary energy [5], by the crystallographic parameters (misorientation angle, rotation axis, and grain-boundary plane) within the coincidence-site lattice model [6], and by the types of grain boundaries, using a combination of the first two approaches [7]. The grain-boundary energy depends on the crystallographic parameters of the boundaries. Low-energy boundaries are characterized by distinct crystallographic parameters and morphological features and termed special boundaries [5–7]. In a real crystal, the low-energy boundaries have slightly different parameters from those of special boundaries and so are called near-special boundaries [8]. These are generally faceted boundaries. Among special boundaries are rectilinear boundaries in section metallographic specimens. The relative number of special boundaries and associated range of $\Sigma$ (the reciprocal of the coincidence-site density) depend on the type of metal or alloy, grain size, and prior thermal history [9–12]. The boundaries whose crystallographic parameters differ drastically from those of special boundaries (the angle of deflection is larger than Brandon’s angle [12]) are referred to as random boundaries [8]. The latter are observed in section metallographic specimens both as curvilinear and rectilinear boundaries [11–12]. Undoubtedly, electron microscopy used for the identification of grain boundaries produces the most reliable results. However, this technique does have limitations associated with the difficulty of collecting macrostatistics, on the one hand, and with a relatively small average grain size, on the other. These problems may be resolved by using an optical method [2, 13] wherein varying etch response of random and special boundaries is used as the basis for the separation of random boundaries from special ones.

We have performed experiments to study the etch response of grain boundaries by a combination of the optical method and electron microscopy. With the majority of electrolytes used, random boundaries exhibited a higher etch response than those of the special type. To illustrate, for Ni$_3$Fe, no matter what is the state of the atomic order, use was made of the foam of a supersaturated solution of chromic anhydrate in orthophosphoric acid. When Ni$_3$Fe with the short-range atomic order was etched in aqua regia (10 mL nitric acid + 10 mL hydrochloric acid), special boundaries showed a higher etch response. Therefore, the classification of grain boundaries according to the degree of their etching should be used with care. This is especially true for rectilinear boundaries. Hence, a combination of optical metallography and electron microscopy appears to be optimal [11–12].

A large body of pertinent information is extracted from a triple-point analysis [14]. At a triple point with random boundaries, the angles between the boundary lines on the plane of the section metallographic specimen, as a rule, approach 120°. At a triple point, where there is one boundary of the special type, the angle lying opposite to the special boundary is close to 180°.

Study of the mutual positions of different types of boundaries found in different materials is an important task [15], because it allows the Hall–Petch relation to be used properly. In the stage of plastic deformation, grain-boundary sliding is known to occur along twin boundaries in fcc crystals [16]. Special and random boundaries have varying grain-to-grain shear...
strength. Along with other factors, it influences the parameters in the Hall – Petch relation [17] which reads: \( \sigma = \sigma_0 + k d^{1/2} \), where \( \sigma_0 \) is the deformation stress inside the grain and \( kd^{1/2} \) is the contribution due to the grain size. In a wide grain-size range, the coefficient \( k \) is a variable quantity [18].

**TEST MATERIAL**

As mentioned above, a polycrystalline Ni₃Fe alloy in the state of short-range atomic order was chosen as the test material. The stacking fault energy for this alloy is 30–40 ergs/cm² [19]. The specimens were heat-treated by homogenizing annealing followed by annealing of the as-forged material and finish annealing at 970 K for 30 min to provide the required grain size. The grain structure was studied by the optical technique. The specimens were subjected to electrolytic polishing (95% acetic acid + 5% perchloric acid) and then etched in the foam of this electrolyte. The etch response appeared to vary with the type of grain boundary: random boundaries were etched to a greater extent than special boundaries.

The grain size distribution was determined by the secant method [20]. About 500 grain sections and the spacing between boundaries of different types were measured (between special boundaries, between random and special boundaries, and between random boundaries). The fractions of grains having only special boundaries, those with both special and random boundaries, or with random boundaries alone, were defined as the ratio of the number of measurements of the spacing between boundaries of a particular type to the total number of measurements. The grains with random boundaries will be called the “parent” grains. These grains are near-polyhedral in shape. We have determined the size of parent grains with outer boundaries of the random type and varying number of inner boundaries of the special type. The fraction of grains with varying number of special boundaries was found as the ratio of the number of these grains to the total number of grains under observation. In the bulk material, the grains with random boundaries are polyhedral in shape. The area of the plane section of grains can be estimated as the area of a circle whose diameter equals the average parent-grain size. The following relation defines the fraction, \( \Delta^k_1 \), of the plane section of the test polycrystal with grains having varying number of special boundaries:

\[
\Delta^k_1 = \frac{\delta_1 d_1^2}{\delta_1 d_1^2 + \delta_2 d_2^2 + \ldots + \delta_k d_k^2},
\]

where \( k \) is the number of special boundaries in a parent grain, \( \delta_k \) is the fraction of parent grains having \( k \) special boundaries, and \( d_k \) is the average size of parent grains with \( k \) special boundaries.

We have estimated the grain-boundary density \( n \) for boundaries of a particular type, using the average spacing between boundaries and the fraction of grains having boundaries of a particular type. The density of special boundaries is thus calculated by the following formula:

\[
n = \frac{\delta_1}{d_1} + \frac{0.5 \delta_2}{d_2},
\]

where \( \delta_1 \) is the fraction of grains with special boundaries, \( d_1 \) is the average spacing between special boundaries, \( \delta_2 \) is the fraction of grains with both random and special boundaries, \( d_2 \) is the average spacing between random and special boundaries, and the coefficient 0.5 accounts for the number of special boundaries in parent grains. In a similar manner, the density of any type of boundaries is determined.

To estimate the grain-boundary energy at equilibrium and quasi-equilibrium triple points, use is made of the well-known relationship between energy and opposite angles at a triple point on the surface of the metallographic specimen

\[
\frac{\gamma_1}{\sin \alpha_1} = \frac{\gamma_2}{\sin \alpha_2} = \frac{\gamma_3}{\sin \alpha_3},
\]

where \( \gamma_1, \gamma_2, \) and \( \gamma_3 \) denote the energy of the grain boundaries at a triple point and \( \alpha_1, \alpha_2, \) and \( \alpha_3 \) are the opposite angles between the grain boundary lines on the plane of the metallographic specimen. According to [17], the maximum energy of random grain boundaries is estimated to be 25% of the free surface energy. The energy is a maximum for a grain boundary with a 90° opposite angle. Then the relative energy, which is the ratio of grain boundary energy to maximum energy of random boundaries, can be estimated from a relation of the form