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INTERACTION OF $Fe_{16}N_2$ NITRIDES IN IRON*

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The distribution of $Fe_{16}N_2$ nitrides was investigated. It was found that in Fe-N samples aged isothermically, the nitrides are, in certain areas, arranged parallel to each other. In samples aged for 0.5 h at a temperature of 528 K and then at 383 K, a characteristic arrangement of small nitrides was observed in the vicinity of the large ones. The results indicate that there is a strong interaction of nitrides through the field of stresses.

1. Introduction

As was established in references [1, 2], during the ageing process of supersaturated Fe–N solutions, magnetic $Fe_{16}N_2$ nitrides are precipitated, with equal probability, on all three planes of the {100} type. On the other hand, Nakada and co-workers [3] have observed that an anisotropic distribution of $Fe_{16}N_2$ nitrides is effectively influenced by external stresses. Thus by applying suitably high external stresses during the ageing process they have caused the precipitation of nitrides only on one plane of the {100} type. A similar effect is also produced by an external magnetic field. In [4] it was observed that if the ageing of samples is carried out in an external magnetic field, the precipitation of $Fe_{16}N_2$ nitrides takes place mainly on {100} planes normal to the direction of the field. However, as shown in [5] that is not an essential condition, a spontaneous arrangement of nitrides has been observed without the interference of an outside agent. Both, in mono- and polycrystalline samples, after a suitably long period of ageing, the nitrides take, in certain areas, positions parallel to each other (they occupy only one of the {100} planes). The mechanism involved in spontaneous arrangement of nitrides is not yet fully explained [5].

The object of this work is a further investigation of the process of spontaneous arrangement of $Fe_{16}N_2$ nitrides in iron.

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2. *Experimental procedure*

The material used in this work was pure iron supplied by the firm Johnson–Matthey. Samples, in the form of strips 0.5 mm thick, were nitrided in mixture of ammonia and hydrogen for 3 hrs at a temperature of 793 K. After nitriding, samples were supersaturated by immersing directly in water. Following that operation the samples were aged for 0.5 h at 528 K and rapidly cooled in water. Samples so treated have been divided into three lots and then additionally aged for 3, 6 and 100 hrs respectively at a temperature of 383 K. Metallographic observations have been carried out with the aid of a Neophot 2 optical microscope, and electronographic examination with a JEM 120 electron microscope at 120 kV.

3. *Results and discussion*

Fig. 1a shows a typical sample surface after nitriding. The presence of Fe_4N nitrides (γ' nitrides) on the surface indicates that nitrogen concentration in the sample is close to equilibrium, as shown by the phase diagram and is about 0.060% weight. Fig. 1b shows the surface of a sample after additional ageing for 0.5 h at 528 K and followed by etching.

Fig. 1a

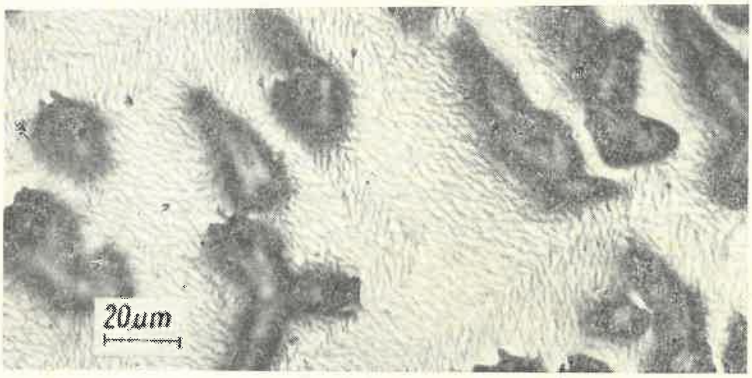


Fig. 1b

Fig. 1c

Fig. 1. The microstructure of iron samples after nitriding in a mixture of ammonia and hydrogen for 3 hrs at 793 K, and the supersaturation (Fig. 1a shows Fe_4N nitrides), and subsequent ageing for 0.5 h at 528 K and cooling (Fig. 1b shows $Fe_{16}N_2$ nitrides). In Fig. 1c is shown the middle layer of the sample presented in Fig. 1b

The $Fe_{16}N_2$ nitrides (α'' nitrides), revealed in Fig. 1b, form areas of habit planes parallel to each other. The magnitude of these areas is smaller on the surface than inside the sample (see Fig. 1c). This shows that the magnitude of these areas depends essentially on the γ' nitrides. Further confirmation of this statement is that in areas of the sample, in which the concentration of γ' nitrides is lower have larger areas with parallel distribution of α'' nitrides. These areas are separated by planes of the {100} type.

Fig. 2a shows the microstructure of a sample aged for 0.5 h at 528 K and subsequently for 3 hrs at 383 K. As follows from the microstructure, in addition to large α'' nitrides that were precipitated during the ageing process at higher temperature, after repeated ageing, numerous small $Fe_{16}N_2$ nitrides have appeared. Small nitrides are precipitated mainly on dislocations (Fig. 2a), but also on edges of large nitrides (Fig. 2b). Small nitrides, which decorate the large ones, are situated on the same crystallographic plane as the large nitrides.

After ageing for 6 hrs at 383 K, numerous precipitations of small nitrides have appeared, which were not connected with dislocations (see Fig. 3a). However, the distribution of these nitrides is not random, and they are mainly arranged at right anglers to the plane of large nitrides, as shown in Fig. 3b. It was also observed that at the ends of large nitrides the small ones are parallel to the plane of large nitrides.

After a prolonged ageing (100 hrs) at 383 K the amount of small nitrides diminishes and they are mainly arranged at right angles to the large ones (Fig. 4). After the extended ageing process there was no evidence of small nitrides adhering to decorate the large ones.

Observed in [5], and confirmed by the present investigations, spontaneous formation by $Fe_{16}N_2$ nitrides of areas parallel to each other, can be, by analogy to the assumptions advanced in [3, 4], attributed to two types of interaction: a) interaction of nitrides with a spontaneous magnetization of iron; b) interaction of nitrides through a stress field. On the other hand, as shown the observations (Figs 1a and 1b), and the results of [5], areas

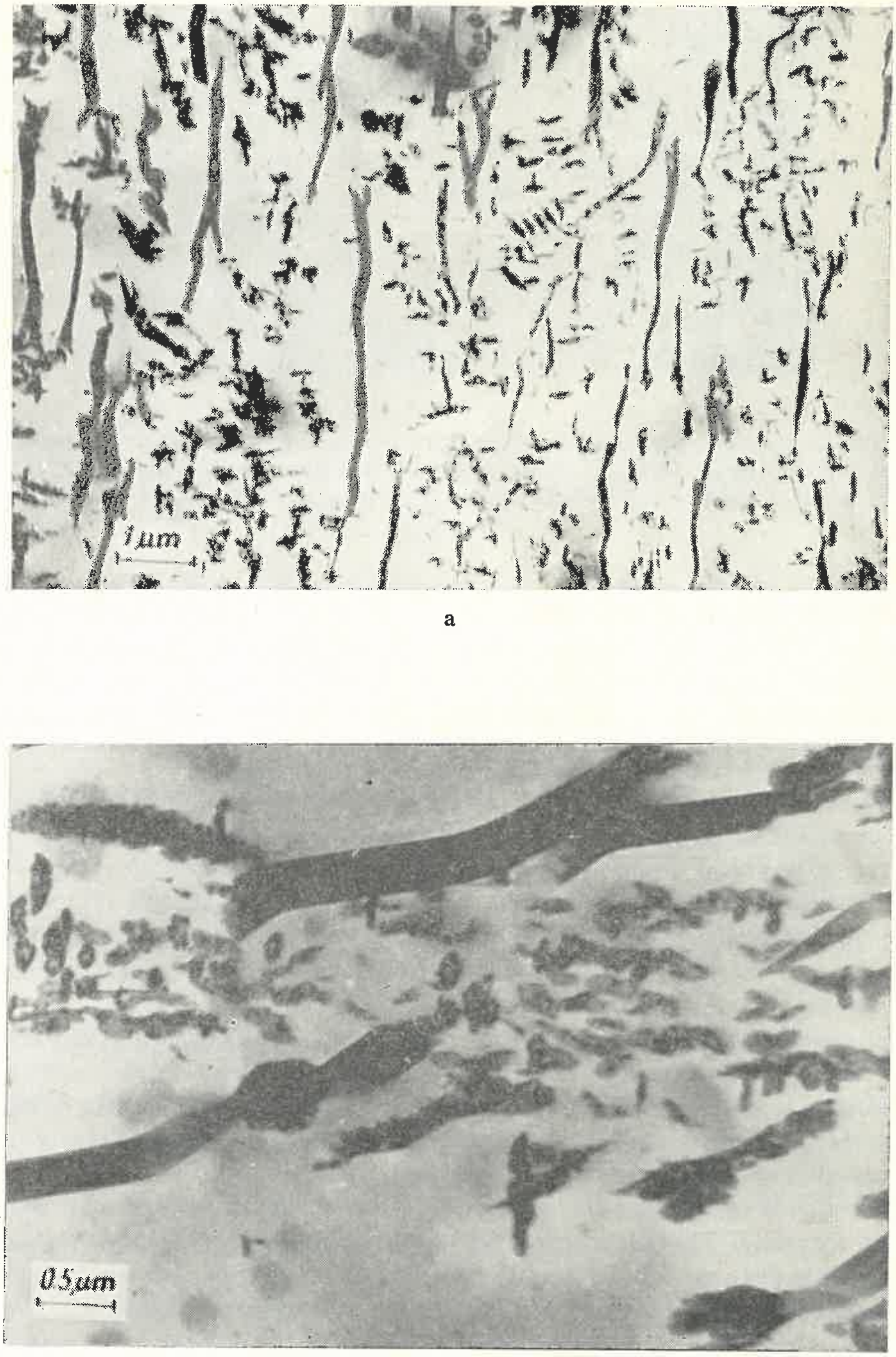
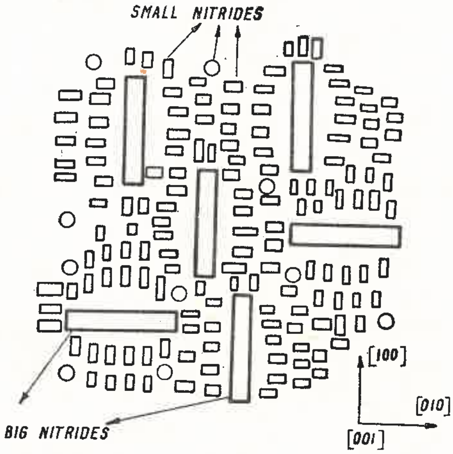


Fig. 2. The microstructures of iron samples aged for 0.5 h at 528 K, and then additionally for 3 hrs at 383 K

a



b

Fig. 3. The microstructure of the iron sample aged for 6 hrs at 383 K and the scheme of nitride distribution

with parallelly arranged nitrides are not similar, i.e. they do not tally with domains observed in iron. That means, that the spontaneous magnetization I_S in iron is of no essential significance in the self-arrangement process of nitrides. The systematically ordered nitride areas are separated by planes of the {110} type (Fig. 1b). The presence of that type of boundaries can be explained by studying the deformation of single areas of ordered nitrides. All these areas are slightly elongated in the direction normal to the plane occupied by

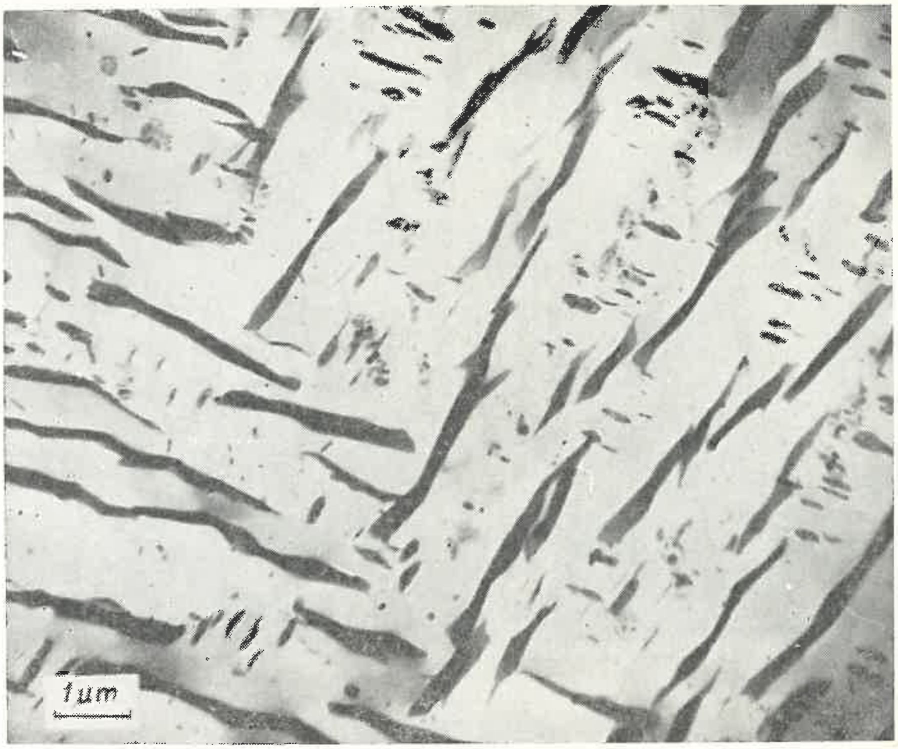


Fig. 4. The microstructure of the iron sample aged for 100 hrs at 383 K

nitrides. (In the region occupied by the nitride ca 10% elongation of the iron lattice takes place in the direction normal to the plane of the nitride [6].) If we consider two adjoining areas, they would fit each other if the plane separating them is of the {110} type. Thus, the {110} type boundaries between the ordered nitrides areas, observed in Figs 1a and 1c, are related to the condition of minimum stress energy.

The phenomenon of the characteristic arrangement of small nitrides in the vicinity of large nitrides, observed in the present work (see Fig. 3), can be also attributed to the interaction through a stress field. If a large precipitation is regarded as a dislocation loop, situated e.g. in the plane (001) with Burgers vector [001], then in the vicinity of its rotation axis, tensile stresses would be of σ_{11} and σ_{22} type. These would cause small nitrides to precipitate on the planes normal to the large surface of large nitrides. Tensile stresses near the ends of large nitrides would, however, be of σ_{33} type, and thus resulting in agreement in the precipitation direction of both large and small nitrides, i.e. they would be situated on the same plane. Should the arrangement of small nitrides depend on spontaneous magnetization, then the precipitation of large and small nitrides should be identical.

Thus, these investigations provide further evidence that the systematic arrangement of $Fe_{16}N_2$ nitrides depends mainly on their interaction through the stress field. This means, that for nitrides of similar size, the stress energy is lower in systems of arranged nitrides being parallel to each other than in the case of random distribution of nitrides.

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