

COERCIVE FIELD MEASUREMENTS ON INHOMOGENEOUS IRON ALLOYS¹⁾

M. FÖLDEAKI²⁾, L. KÖSZEGI³⁾, Budapest

It is shown that changes in the coercive field of Fe-Me alloys (Me = Al, Si, Nb, Ti, Zr) with increasing Me-concentration can be well correlated to the solid solubilities and nucleation behaviour of Me. Me atoms, forming solid solutions with Fe, do not influence the coercive field significantly, whereas H_c increases sharply if Me precipitates incoherently in the matrix or at the dislocations.

I. INTRODUCTION

Often it is not easy to predict the microstructure of an iron alloy corresponding to certain heat treatments, although this knowledge is very useful for the development of outstanding metallurgical material properties. Ferromagnetic properties are in general highly structure-sensitive. Therefore, coercive field (H_c) measurements may give valuable qualitative information on the microstructure [1, 2], supplementing or replacing transmission electron microscopy. In the present study, H_c and microstructural investigations were carried out on a great number of Fe—(0.02 wt %—3 wt %)Me alloys (Me = Al, Si, Ti, Nb, Zr), after different annealing and nitriding treatments. The substitutional annealing elements were selected to ensure a broad solid solubility range [3, 4].

II. SAMPLE PREPARATION

The alloys were melted from high purity Fe and Me in an electron beam furnace and cold swaged to wires of 1 mm \varnothing . Several different thermal pre-treatments were carried out in dry H_2 : i) anneal at 1000 K, cooling rate 100 K/hour; ii) anneal

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²⁾ Institute for Materials Testing and Quality Control of the Engineering Industry, BUDAPEST, Hungary.

³⁾ Central Research Institute for Physics, BUDAPEST, Hungary.

at 1273 K, cooling rate 100 K/hour; iii) anneal at 1373 K, cooling rate 2 K/sec. (quenching). Nitrogen was introduced by annealing the specimens during 4—36 h in 2.5% $\text{NH}_3 + \text{H}_2$ mixtures at 673 K or 773 K.

III. EXPERIMENTAL RESULTS AND DISCUSSION

III.1. Microstructure

The microstructure before nitriding depends on the Fe—Me phase diagram considering ferromagnetic interactions [3, 4] and on the nucleation properties of the alloy [5]. Consequently, Me may be presented as a substitutional solute or as precipitate, nucleated on dislocations or in the matrix.

After nitriding, nitrogen may be present as interstitial solute in the matrix or in the strain field of precipitates or dislocations, or as a component of nitride precipitates (MeN or mixed Fe—Me—N clusters). Nitride precipitates may nucleate again in the matrix or on dislocations.

The microstructure of Fe—Al—N, Fe—Ti—N and Fe—Zr—N alloys was investigated by transmission electron microscopy [6, 7, 8]. No precipitation could be observed in Fe—Al—N excepted the sample cold-worked prior to nitriding [6]. Plate-like precipitates dominate in the Fe—Ti—N alloys [8], whereas only precipitation on dislocations could be observed in Fe—Zr—N [7].

III.2. Coercive field measurements

The coercive field was measured using an automated Förster coercimeter.

Whereas the total H_c value of a single sample cannot be interpreted quantitatively in terms of the microstructure it is more easy to relate H_c to well defined changes of the microstructure. As shown in Fig. 1, no concentration dependence of H_c on Me was observed on annealed Fe—Al samples, whereas the coercive field of the deformed samples increases significantly with Al content due to enhanced precipitation of AlN on dislocations. Fig. 2 shows the change of H_c with composition for different substitutional solubilities. For good (Si) or practically zero (Nb, Zr) solid solubilities no significant influence of thermal pre-treatment could be detected. The measured H_c values of Fe—Ti or Fe—Ti—N alloys fluctuate randomly between 0.5 and 1 Oe for low concentrations in Ti (≤ 2 wt %). Significant changes of H_c due to different annealing treatments are found only above the metallurgical solubility limit (Ti > 2 wt %). As it is known from theory [1, 2], H_c depends strongly on the concentration of precipitates and dislocations in the sample: No significant changes occur with fine precipitates ($\leq 0.5 \mu\text{m}$), whereas with coarsening H_c increases sharply and finally saturates [1]. With plastic deformation H_c increases

monotonously [2]. If both lattice defects, dislocations and precipitates are simultaneously present, the increase of H_c is even more pronounced.

On this basis the experimental results presented in Fig. 1 and Fig. 2 may be analysed to give the necessary qualitative information on the microstructure of the investigated systems. The results of such an analysis, using the data on the nucleation behaviour of the substitutional addition as well [5], are summarized in Table 1.

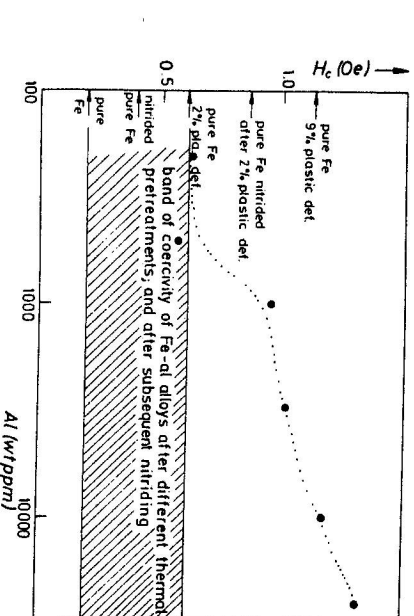


Fig. 1. Change of H_c with Al content in nitrided Fe—Al alloys, annealed (////) or cold worked (●) prior to nitriding. Characteristic values of pure Fe are given as well.

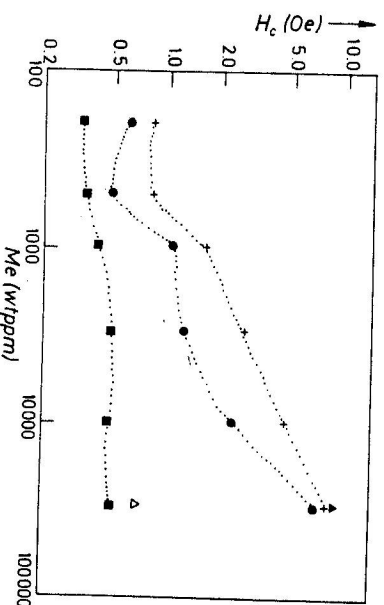


Fig. 2. Change of H_c with Me—content after quenching from 1000°C; Me = Zr (+), Nb (●), Si (■), Ti (Δ) or after annealing at 720 °C; Me = Ti (▲).

Table 1

Correlation of microstructure and H_c

substitutional addition (Me)	pre-treatment	H_c change with Me-concentration	corresponding microstructure
Al, Si	i/ii	negligible/slight increase	random solid solution (1)
Al, Si; Ti \leq 2wt %	iii	slight increase	(1) + dislocations due to quenching
Ti \leq 2wt %	i/ii	negligible/slight increase	fine precipitates in matrix
Ti > 2wt %	i/ii	sharp increase	coarse precipitates in matrix
Nb; Zr \leq 500 wt ppm	i/ii/iii	negligible/slight increase	fine precipitates at disloc.
Nb; Zr > 500 wt ppm	i/ii/iii	sharp increase	coarse precipitates at disloc.

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ИЗМЕРЕНИЯ КОРРПТИВНОЙ СИЛЫ НА НЕОДНОРОДНЫХ СПЛАВАХ ЖЕЛЕЗА

В работе показано, что изменения коррптивной силы сплавов типа Fe—Me (Me = Al, Si, Nb, Ti, Zr) при увеличении концентрации Me могут быть связаны с растрояемостью Me и характером образования активных центров. Атомы металлов Me, образующие с железом твердые растворы, не влияют заметно на коррптивную силу, в то время как H_c увеличивается резко, если Me в матрице или на дислокациях осаждаются некогерентно.