

Dependence of magnetic domain structure on grain orientation in 3.2% silicon-iron with the (1 1 0) [0 0 1] texture

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A series of ferromagnetic domain structures in individual grains of (1 1 0) [0 0 1] textured 3.2% silicon-iron has been observed and measured together with the orientation of the grain. It has been shown that the domain dimensions are inversely proportional to the angle of misorientation of the (1 1 0) plane from the grain surface. This is in very good agreement with the theory given by Špaček [1, 2]. The lozenge domain structure has also been investigated as a function of grain orientation and the measurements are in excellent agreement with the theory proposed by Goodenough [5].

1. Introduction

The ferromagnetic domain structures of grain-orientated (1 1 0) [0 0 1] 3.2% silicon-iron have been investigated in considerable detail [1–4]. The first investigation of the relationship between domain structure and crystallographic orientation in the (1 1 0) [0 0 1] textured material was carried out by Paxton and Nilan [3]. They listed five basic domain structures and concluded that the domain structure in a particular grain depended only on the crystallographic orientation of that grain and was not influenced by the orientation of neighbouring grains. The present investigation is concerned with the relationship between the domain dimensions and grain orientation in order to verify quantitative relationships derived by Špaček [1, 2] and Goodenough [5].

2. Specimen preparation and observation of domain structures

The grain orientated commercial grade material was supplied in the form of a thin strip 3 cm wide and 0.035 cm thick. The (1 1 0) [0 0 1] texture was produced by the conventional multistage rolling process followed by a final anneal at 1150°C in dry hydrogen. The specimens were approximately 3 cm × 1 cm × 0.035 cm and were carefully polished through various grades of silicon carbide paper and finally with 1 μm diamond paste. The polishing was carried out parallel to the rolling direction, i.e. approximately parallel to the

easy direction of magnetization [0 0 1] so as to avoid stray flux at residual scratches.

The specimens were then electro-polished in a solution of 25 g chromium trioxide, 133 ml glacial acetic acid and 7 ml of water. The electrolyte was contained in a stainless steel beaker surrounded by ice. The specimen was used as the anode and the beaker as the cathode. A potential difference of 25 V was applied to the electrodes so as to produce a current density of about 0.1 to 0.2 A cm⁻². It was found essential to maintain a constant temperature during the electro-polishing process and to stir the solution continuously. If the temperature was allowed to rise above 30°C severe etching of the specimen was produced and the electro-polishing properties of the solution irreversibly destroyed. The best polishing conditions were found in the temperature range 15 to 20°C. The specimens were polished for about 20 to 30 min after which they were thoroughly washed and dried. Apart from the thermal instability of the polishing solution it is also highly susceptible to degradation due to prolonged storage, so that fresh electrolyte was made just before use.

A small drop of colloidal magnetite [6] was applied to the surface of the specimen and spread to an even thickness by placing a thin cover slip over it. It was found that better contrast of the domains was obtained if the cover slip was then removed, thus allowing the colloid to dry. The

observations were made on both Zeiss and Reichert metallographic microscopes.

The orientation of individual grains, defined in terms of the angles ϕ and θ was determined by the Laue X-ray back-reflection technique. The definition of the angles ϕ and θ with respect to the rolling plane and the rolling direction was the same as that used by Paxton and Nilan [3]. That one of the $\langle 100 \rangle$ directions which has the smallest deviation from the rolling plane (1 1 0) is referred to as the $[001]$ direction. The angle ϕ is defined as the angle by which the $[001]$ direction deviates from the rolling plane. The angle θ is the angle formed by the rolling direction and the projection of the $[001]$ direction onto the rolling plane.

3. Results

Examples of some of the less well known and more complicated domain structures are shown in Figs. 1 to 5. The rolling direction is in all cases parallel to the vertical edge of the photographs. The easy direction of magnetization is easily identified since even in relatively complex structures such as the lace structure of Fig. 5 there is a preferred orientation and a symmetry of the pattern with respect to the easy direction which usually makes an angle of less than 10° with the rolling direction. For example, in Fig. 5 although the easy direction is not in the plane of the grain, the projection of the easy $[001]$ direction onto the surface of the grain makes an angle of 8° with

rolling direction. It is also interesting to note that in Fig. 3 the domain structure appears to be a mixture of the lozenge and lace structure although the angle ϕ is 6° . This suggests that the pattern classification according to the value of ϕ is approximate only. There is still, however, a noticeable symmetry in the pattern, in particular the projection of the easy direction onto the surface plane makes an angle of $\theta = 6^\circ$.

4. Comparison of results with Špaček's theory

Špaček [1, 2] has shown by a detailed analysis that the domain spacing on the grain surfaces depends very strongly on the grain orientation. The experimentally observed structure is that which minimizes the magnetostatic energy, the anisotropy energy and the wall energy. The full analysis would require the inclusion of the magnetoelastic energy but this would make the mathematics extremely difficult. Using the definition of the orientation angle ϕ given above, the domain spacing, D is given by:

$$D = \left(\frac{12\pi L}{4M_s^2 + K_1} \right)^{\frac{1}{2}} \frac{1}{\phi} \quad (1)$$

where L is the diameter of the grain, M_s the saturation magnetization and K_1 the first magneto-crystalline anisotropy constant. Thus a graph of D versus ϕ^{-1} should be linear with a positive slope. It is, therefore, of considerable interest to check whether this relationship is really valid. The



Figure 1 Simple 180° domain walls, $\phi = 1^\circ$. Rolling direction vertical.

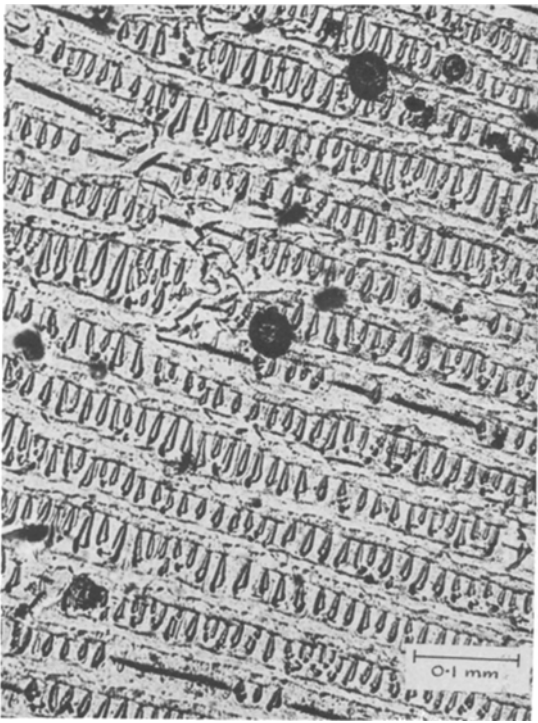


Figure 2 Lozenge domains, $\phi = 6.5^\circ$ "Goodenough walls" visible. Rolling direction vertical.

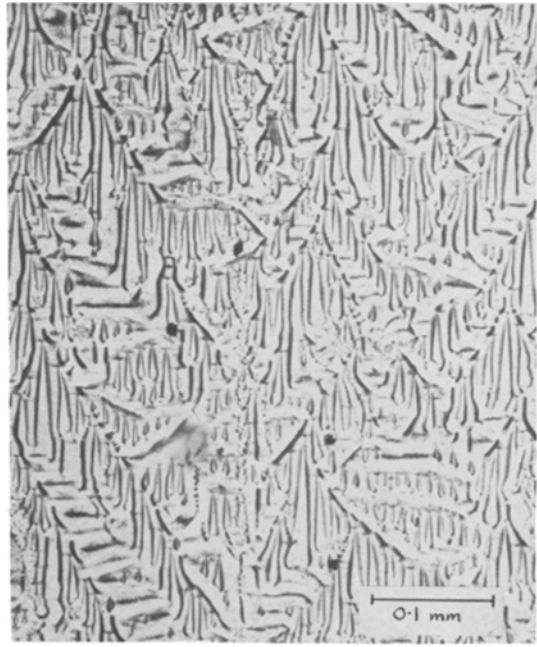


Figure 3 Mixed lace and lozenge structure, $\phi = 6^\circ$, $\theta = 6^\circ$. Rolling direction vertical.

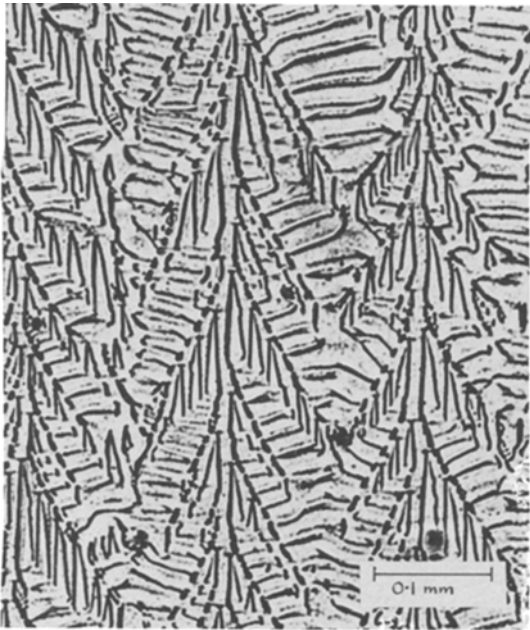


Figure 4 Lace structure, $\phi = 7^\circ$, $\theta = 0$. Rolling direction vertical.



Figure 5 Lace structure, $\phi = 8^\circ$, $\theta = 8^\circ$. Rolling direction vertical.

principal uncertainty lies in the determination of D since the domain structure is not, in general, a simple geometrical pattern. In the case of parallel 180° walls as shown in Fig. 1, D can be taken as the average spacing. However, in the case of the lozenge domain structure shown in Fig. 2 it is clear that the structure is more finely divided though it is not possible to give a precise value for D . In this situation the domain dimensions have to be described by some kind of average representative dimension. This measurement will of course be rather subjective but measurements made by several independent workers show a high degree of consistency and correlation. Measurement of ϕ is relatively simple and was carried out by means of the usual back-reflection Laue technique. The results of measurements on 48 grains are shown in Fig. 6 together with a regression line of D on ϕ^{-1} . The experimentally observed slope is 3.94×10^{-4} and the intercept on the ϕ^{-1} axis is 6 rad^{-1} . A theoretical value for the slope was calculated from the following values of the parameters, M_s , K_1 and L : $M_s = 1600 \text{ e.m.u. cm}^{-3}$, $K_1 = 1.3 \times 10^5 \text{ erg cm}^{-3}$, $L = 0.035 \text{ cm}$, giving slope $m = 3.58 \times 10^{-4}$. This result is not particularly sensitive to the value of K_1 but depends very markedly on M_s which is, fortunately, known with considerable accuracy. In any case $M_s^2 \gg K_1$ so that the denominator is determined largely by the

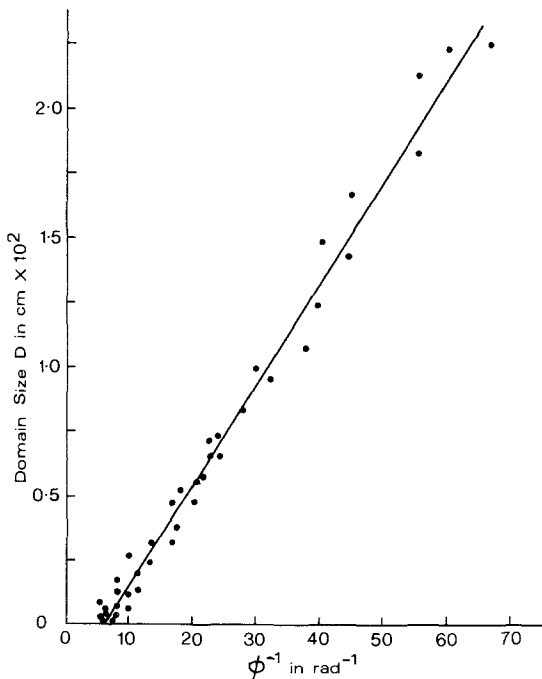


Figure 6 Domain spacing versus reciprocal of orientation angle, ϕ .

numerical value of M_s . The value of L has to be a representative value of the grain size and in view of the importance of the magnetostatic energy in determining the domain structures it was considered that the best choice would be the grain thickness rather than an average value of the grain size in the plane of the sheet. Thus L was taken as 0.035 cm , a dimension which is common to all grains since these almost invariably occupied the whole thickness of the strip. The agreement between the theoretical and experimental results is extremely good and suggests that the Špaček theory has a sound physical basis. This result may be of considerable practical value in the determination of the quality and degree of preferred orientation in textured sheets of Si-Fe since the orientations of large numbers of grains can be determined by a single observation of the domain structures.

5. Lozenge domain structures and comparison with Goodenough's theory

A very common domain structure observed in Si-Fe with the $(110) [001]$ texture is that known as a lozenge structure (Fig. 2). This structure usually appears when the surface plane is orientated such that ϕ is between 4° and 7° so that there is a substantial emergence of flux at the surface resulting in an increase in the magnetostatic energy. The lozenge domains are of opposite magnetic sign to the surface flux so that their appearance reduces the magnetostatic energy by producing short path flux return loops. The lozenges are usually of uniform size and arranged in regularly spaced rows parallel to the $[1\bar{1}0]$ direction as shown in Fig. 7. When an external field is applied parallel to the $[001]$ direction domains enclosing alternate rows of lozenges grow while those in between diminish, the lozenges themselves also grow or shrink in step with the main domains. In order to explain this behaviour Goodenough [5] proposed the existence of "invisible" plane parallel walls between the

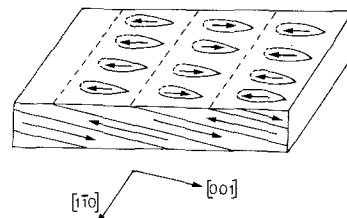


Figure 7 Lozenge structure according to Goodenough.

TABLE I

ϕ (deg.)	D , experimental (cm $\times 10^3$)	D , theoretical (cm $\times 10^3$)	ϕ (deg.)	D , experimental (cm $\times 10^3$)	D , theoretical (cm $\times 10^3$)
5.0	3.53	3.48	6.5	2.55	2.35
5.0	3.35	3.48	6.5	2.35	2.35
6.0	2.50	2.62	6.5	2.55	2.35
6.0	2.50	2.62	6.5	2.35	2.35

rows of lozenges, shown as dotted lines in Fig. 7. By means of a detailed mathematical analysis Goodenough has shown that the spacing D between the plane parallel walls is given by:

$$D = \frac{1}{M_s \sin \phi} \left[\frac{\gamma h}{1.7 \sin \phi} \right]^{\frac{1}{2}} \quad (2)$$

where M_s is the saturation magnetization, ϕ the orientation angle as defined earlier, γ the wall energy and h the thickness of the sheet. The theoretical values of D , as determined from Equation 2, have been compared with the experimental values of D obtained from eight lozenge structures and are shown in Table I. The theoretical value of D for the various values of ϕ were calculated assuming $h = 0.035$ cm, $M_s = 1600$ e.m.u. cm^{-3} and $\gamma = 1$ erg cm^{-2} . The agreement between the experimental and theoretical values of D is very good indeed and suggests that Goodenough's theory has a sound physical basis. The fact that most of the lozenges are open ended also confirms Goodenough's explanation of this in terms of the small angles which the spin vectors in the domain wall make with the specimen surface so that the

flux leakage is small and does not attract the colloidal particles to any significant extent. Since the lozenge structure appears only when the angle ϕ is in the range 4 to 7° it is difficult to investigate the conditions for the state of closure of the lozenges. Further confirmation of Goodenough's theory is provided by the observation that in some cases the "invisible" 180° domain walls parallel to the $[1\bar{1}0]$ direction between the lozenge rows are sometimes just visible, as in Fig. 2.

Acknowledgement

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