

# ON THE USE OF WEDGE-SHAPED SINGLE-CRYSTALLINE SAMPLES IN MEASURING THE THICKNESS-DEPENDENCE OF THE DOMAIN WIDTH IN FERROMAGNETS

BY S. SZYMURA

Magnetic Materials Laboratory, Institute of Ferrous Metallurgy, Gliwice\*

(Received May 20, 1971)

Recent experiments on Co and FeSi single crystals showed that the thickness-dependence of the domain width in ferromagnets can be determined quite accurately from measurements on specific wedge-shaped samples. In the present paper, we examine the influence of the wedge angle and the crystallographic orientation of a sample's slanted faces on such measurements. In particular, we study the modified Landau-Lifshitz domain structure in FeSi within the thickness interval from  $10^{-3}$  cm to 1 cm. It is shown that the wedge angle has little influence on the measurements if the sample's crystallographic orientation is symmetric. Otherwise the thickness-dependence curve of the domain width shifts markedly upwards with increasing wedge-angle, although the power-laws governing the curve below and above the critical thickness  $T_0$  remain unchanged.

## 1. Introduction

In [1] the dependence of domain width  $D$  on crystal thickness  $T$  along a magnetically preferred direction was determined experimentally for uniaxial cobalt within a broad range of thicknesses, from  $2 \times 10^{-4}$  cm to  $3 \times 10^{-1}$  cm. The regular honeycomb and Goodenough remanent domain structures were considered. These studies were performed on a wedge-shaped single-crystalline sample with an angle of inclination of  $18^\circ$  and wedge base perpendicular to the magnetically preferred [0001] direction.

In [2] the  $D(T)$  dependence obtained thus was compared with the results of measurements made on a series of rectangular-prism-shaped samples of various thicknesses along the magnetically preferred direction. The agreement was found to be very good.

Basing on the results of [1, 2], the same method was applied [3, 4] in determination of the  $D(T)$  dependence in the  $10^{-3}$  cm to  $10^{-1}$  cm thickness range for two domain structures in three-axial FeSi, namely, the simple (SL) and modified (ML) Landau-Lifshitz domain structures. In this case use was made of wedge-shaped samples with angles of inclination of  $20^\circ$  and  $18^\circ$ , respectively, and bases perpendicular to the  $\langle 100 \rangle$

\* Address: Zakład Materiałów Magnetycznych, Instytut Metalurgii Żelaza, Gliwice, K. Miarki 12, Poland.

preferred direction. For the *ML* domain structure the results of [3, 4] were verified in [5] by measurements on a series of appropriately crystallographically oriented parallelepiped samples. Once again it proved that the agreement between the  $D(T)$  dependences determined by both methods is satisfactory, although somewhat worse than in the case of cobalt. Moreover, in [5] there is the first ever analysis of the influence of the crystallographic orientation of a rectangular-prism-shaped single-crystalline sample on the  $D(T)$  dependence for the *ML* domain structure in FeSi. It was found that the general power law

$$D_{X,m} = a_{X,m} T^{b_m} \quad (1)$$

given in [4], remains valid, but the sample's crystallographic orientation bears an effect on the coefficient  $a_{X,m}$  only. In Eq. (1) the subscript  $X$  denotes the type of domain structure, whereas  $m = 1$  for  $T \leq T_0$  and  $m = 2$  for  $T \geq T_0$ , where  $T_0$  is the so-called critical thickness. In particular, crystallographic orientation does not alter the value of critical thickness  $T_0$ , which for FeSi is  $4 \times 10^{-2}$  cm (cf. [3, 4]).

One of the most important results of [1-5] is the conclusion that the tedious and expensive determinations of the  $D(T)$  relation with the use of a series of single-crystalline samples of various thicknesses can be successfully accomplished by much simpler measurements on a single wedge-shaped sample. The data in [5] show, however, that results of measurements of  $D(T)$  may depend on the angle of inclination and the crystallographic orientation of the slanted faces of the wedge-shaped sample. The aim of the present paper is to explain this problem on the example of *ML* domain structure in FeSi.

## 2. Observations and measurements

Investigations employed eight Fe-3.25%Si single crystals cut into wedges, the dimensions  $w$ ,  $t$  and crystallographic orientation of which are presented in Fig. 1. The various samples differed by the angles  $\vartheta$  and  $\theta$ . Namely, with respect to crystallographic orientation four samples were so-called "one-sided" wedges ( $\vartheta = 0$ ;  $\theta = 18^\circ, 23^\circ, 40^\circ$  and  $45^\circ$ ), two were asymmetrical wedges ( $\vartheta = 10^\circ, 22^\circ$  and  $\theta = 30^\circ, 28^\circ$ , respectively), while the remaining two were symmetrical wedges ( $\vartheta = \theta = 12.5^\circ$  and  $18^\circ$ ).

The single crystals were obtained from a polycrystalline sheet metal band by the method of secondary recrystallization described in [6], which resides in pulling the band at a certain speed through a heating device with large temperature gradient. The accuracy in determining the crystallographic orientation of the (110) sides of the samples was  $\pm 10'$  (Kristalloflex 4, Siemens).

The mechanical and electrolytic polishing of the samples and the colloid technique used in determining the domain structure were the same as in [7-10]. For the observations a Neophot 2 (Zeiss) metallographic microscope was used, and the distance  $d$  between the Bloch lines on the (110) surface of the samples, as well as the crystal thicknesses  $T$  and the angles  $\vartheta$ ,  $\theta$  were determined from averaged measurements on large photographs of the sample and the domain pattern. To account for incidental distortions of the domain structure (local stresses, large vacancies or impurities, etc.) the samples were heated

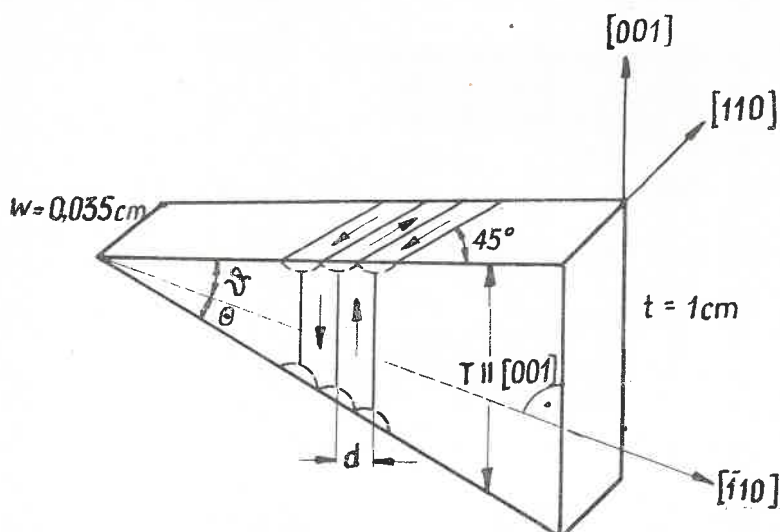


Fig. 1. The shape, dimensions and crystallographic orientation of samples used in experiments

above the Curie temperature and cooled down to room temperature several times and the observations and measurements were repeated. In this way, satisfactory accuracy in the  $d$ ,  $T$ ,  $\vartheta$  and  $\theta$  measurements was achieved, and the error for any of these quantities did not exceed 5% for small and 3% for large thicknesses ( $T > 10^{-2}$  cm).

### 3. Results of measurements

Typical powder patterns observed on the (110) crystal surfaces are shown in Figs 2 and 3. The patterns in Figs 2a, b correspond to "one-sided" wedges ( $\vartheta = 0$  and  $\theta = 23^\circ$  and  $45^\circ$ , respectively), that in Fig. 2a to the symmetrical wedge ( $\vartheta = \theta = 12.5^\circ$ ), whereas those of Figs 3b, c to the asymmetrical wedge ( $\vartheta = 22^\circ$  and  $\theta = 28^\circ$ ). Figure 3b comprises the range of thicknesses  $T$  up to  $1.6 \times 10^{-2}$  cm, whereas Fig. 3c illustrates the  $ML$  domain structure on the same sample for  $T > 1.75 \times 10^{-1}$  cm.

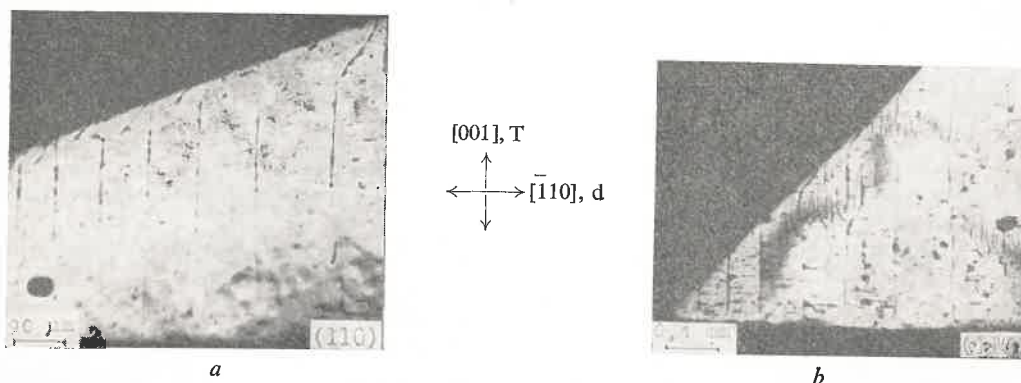


Fig. 2. Powder patterns on (110) crystal surfaces showing the modified Landau-Lifshitz ( $ML$ ) domain structure: a)  $\vartheta = 0, \theta = 23^\circ$  and b)  $\vartheta = 0, \theta = 45^\circ$

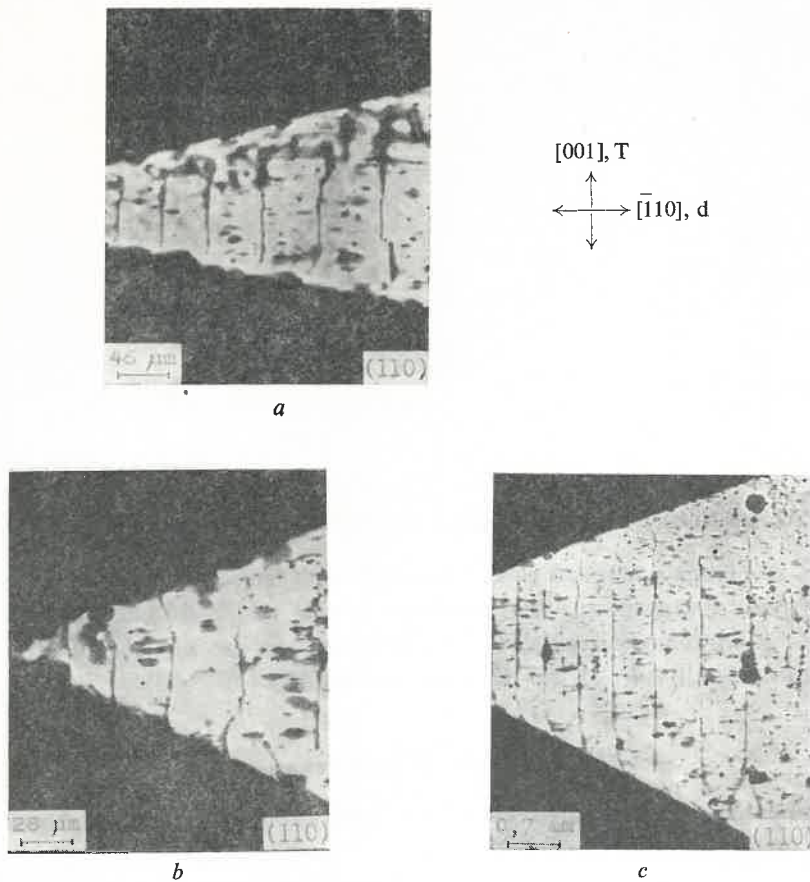


Fig. 3. Powder patterns on (110) crystal surfaces a)  $\theta = \theta = 12.5^\circ$ , and b) and c)  $\theta = 22^\circ$ ,  $\theta = 28^\circ$ ; Figs b, c are patterns from the same sample and include the thickness range for  $T < 1.6 \times 10^{-2} \text{ cm} < T_0$  b) and  $T > 1.75 \times 10^{-1} \text{ cm} > T_0$  c)

By way of illustration Fig. 4 presents by means of the notation  $D_{ML}^{\theta;\theta}$  the curves or measurement points for five of the eight examined samples.

Readings taken from large plots of the  $D(T)$  curves lead to the following values for all curves

$$b_1 = 0.5, b_2 = 0.9 \text{ and } T_0 = 4 \times 10^{-2} \text{ cm} \quad (2)$$

in accordance with the results of [3-5].

For the coefficients  $a_{ML;m}^{\theta;\theta}$  defined by Eq. (1) the readings gave the following values:

$$\begin{aligned} a_{ML;1}^{12.5;12.5} &= 0.0293, & a_{ML;1}^{18;18} &= 0.0296, \\ a_{ML;1}^{22;28} &= 0.0300, & a_{ML;1}^{0;18} &= 0.0300, \\ a_{ML;1}^{0;23} &= 0.0305, & a_{ML;1}^{10;30} &= 0.0310, \\ a_{ML;1}^{0;40} &= 0.0335, & a_{ML;1}^{0;45} &= 0.0340 \end{aligned} \quad (3)$$

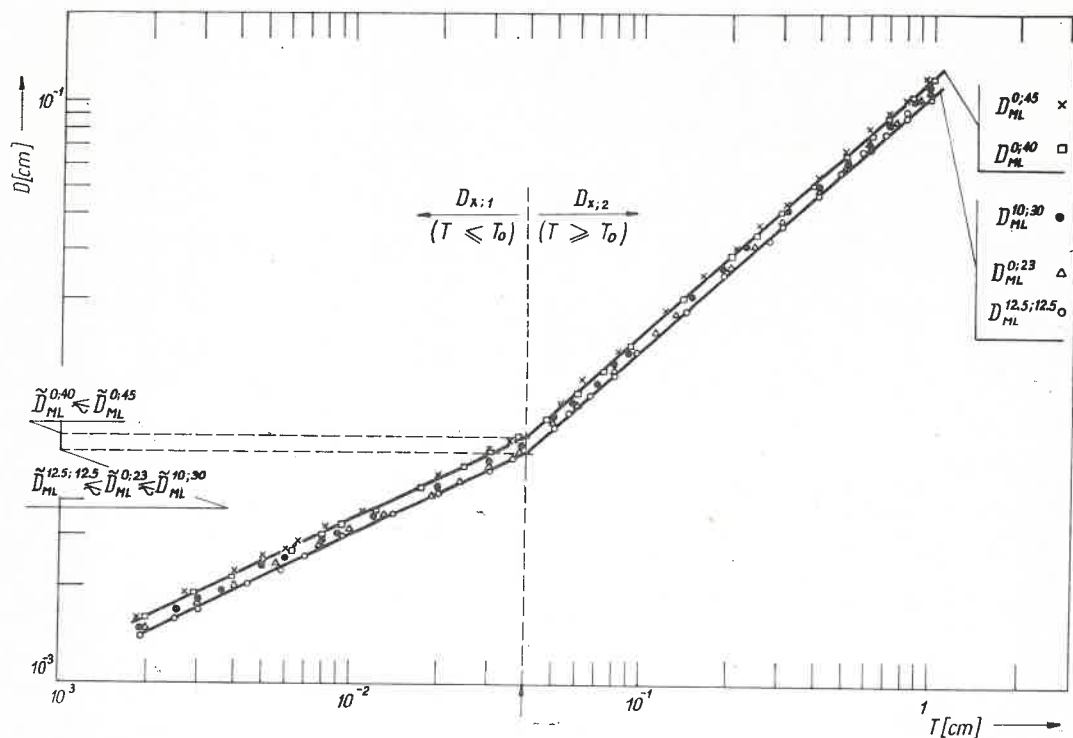


Fig. 4. Thickness-dependence curves of the domain widths for modified domain structure of wedge-shaped Fe-3.25% Si single crystal for:  $\vartheta = \theta = 12.5^\circ$  ( $D_{ML}^{12.5;12.5}$  curve);  $\vartheta = 0, \theta = 23^\circ$  ( $D_{ML}^{0;23}$  curve);  $\vartheta = 10^\circ, \theta = 30^\circ$  ( $D_{ML}^{10;30}$  curve);  $\vartheta = 0, \theta = 40^\circ$  ( $D_{ML}^{0;40}$  curve);  $\vartheta = 0, \theta = 45^\circ$  ( $D_{ML}^{0;45}$  curve)

for  $m = 1$  ( $T \leq T_0$ ), and

$$\begin{aligned}
 a_{ML;2}^{12.5;12.5} &= 0.1065, & a_{ML;2}^{18;18} &= 0.107, \\
 a_{ML;2}^{22;28} &= 0.1100, & a_{ML;2}^{0;18} &= 0.110, \\
 a_{ML;2}^{0;23} &= 0.1105, & a_{ML;2}^{10;30} &= 0.112, \\
 a_{ML;2}^{0;40} &= 0.1210, & a_{ML;2}^{0;45} &= 0.124
 \end{aligned} \quad (4)$$

for  $m = 2$  ( $T \geq T_0$ ), in  $\text{cm}^{1-b_m}$  units.

A check of the correctness of the readings (3) and (4) is the constancy of the ratio

$$a_{ML;1}^{9;0} / a_{ML;2}^{9;0} = 0.276 \text{ cm}^{0.4}. \quad (5)$$

Its value is in agreement with the results obtained in [3-5].

The critical domain widths,  $\tilde{D}_{ML}^{\vartheta;0}$ , corresponding to the critical thicknesses  $T_0$  computed from the equation

$$\tilde{D}_{ML}^{\vartheta;0} = [(a_{ML;1}^{\vartheta;0})^{b_2} (a_{ML;2}^{\vartheta;0})^{-b_1}]^{\frac{1}{b_2 - b_1}} \quad (6)$$

TABLE I

Critical crystal thickness $T_0$		400 $\mu\text{m}$										Rectangular-prism-shaped sample in [5] ( $\phi = 0$ )				
		Wedge-shaped samples														
Angles	$\phi$	12.5°		18°		23°		30°		45°		0°				
		$T \leq T_0$ (m=1)	$T \geq T_0$ (m=2)	$T \leq T_0$ (m=1)	$T \geq T_0$ (m=2)	$T \leq T_0$ (m=1)	$T \geq T_0$ (m=2)	$T \leq T_0$ (m=1)	$T \geq T_0$ (m=2)	$T \leq T_0$ (m=1)	$T \geq T_0$ (m=2)					
Crystal thickness $T$		0.5	0.9	0.5	0.9	0.5	0.9	0.5	0.9	0.5	0.9	0.5	0.9	$T \leq T_0$ (m=1)	$T \geq T_0$ (m=2)	
Exponent $b_m$		0.5	0.9	0.5	0.9	0.5	0.9	0.5	0.9	0.5	0.9	0.5	0.9	0.5	0.9	
Coefficient $a_{ML;m} [\text{cm}^{1-b_m}]$		0.0293 $\text{cm}^{0.5}$	0.1065 $\text{cm}^{0.9}$	0.0296 $\text{cm}^{0.5}$	0.107 $\text{cm}^{0.9}$	0.03 $\text{cm}^{0.5}$	0.110 $\text{cm}^{0.9}$	0.031 $\text{cm}^{0.5}$	0.112 $\text{cm}^{0.9}$	0.034 $\text{cm}^{0.5}$	0.124 $\text{cm}^{0.9}$	0.029 $\text{cm}^{0.5}$	0.106 $\text{cm}^{0.9}$	0.029 $\text{cm}^{0.5}$	0.106 $\text{cm}^{0.9}$	
Critical domain width $\tilde{D}_{ML}^{\phi, \theta} (T_0) = a_{ML;m} T^{b_m}$		58.6 $\mu\text{m}$										61 $\mu\text{m}$	62 $\mu\text{m}$	68 $\mu\text{m}$	58 $\mu\text{m}$	
$a_{ML;1} / a_{ML;2} = T^{b_2 - b_1} [\text{cm}^{b_2 - b_1}]$		0.276 $\text{cm}^{0.4}$														
$\tilde{D}_{ML;m}^{12.5;12.5} = \tilde{D}_{ML}^{12.5;12.5}$		1		1.01		1.04		1.06		1.17		1.06		1.17		0.99
$\tilde{D}_{ML;m}^{18;18} = \tilde{D}_{ML}^{18;18}$		0.99		1		1.02		1.04		1.14		1.04		1.14		0.99
$\tilde{D}_{ML;m}^{\phi, \theta; 23} = \tilde{D}_{ML}^{\phi, \theta; 23}$		0.97		0.97		1		1.02		1.12		1.02		1.12		0.96
$\tilde{D}_{ML;m}^{\phi, \theta; 30} = \tilde{D}_{ML}^{\phi, \theta; 30}$		0.95		0.96		0.98		1		1.09		1		1.09		0.93
$\tilde{D}_{ML;m}^{\phi, \theta; 45} = \tilde{D}_{ML}^{\phi, \theta; 45}$		0.86		0.87		0.86		0.91		1		0.91		1		0.85



are (in  $10^{-4}$  cm units):

$$\begin{aligned}
 \tilde{D}_{ML}^{12.5;12.5} &= 58.6, & \tilde{D}_{ML}^{18;18} &= 59.5, \\
 \tilde{D}_{ML}^{22;28} &= 60, & \tilde{D}_{ML}^{0;18} &= 60, \\
 \tilde{D}_{ML}^{0;24} &= 61, & \tilde{D}_{ML}^{10;30} &= 62, \\
 \tilde{D}_{ML}^{0;40} &= 67, & \tilde{D}_{ML}^{0;45} &= 68.
 \end{aligned} \tag{7}$$

Taking into account the values (3) and (4) of the  $\alpha_{ML;m}^{\vartheta;0}$  coefficients, the dependence (1) of domain width on crystal thickness, in the examined wedge-shaped FeSi samples with  $ML$  domain structure, takes the following form:

$$\begin{aligned}
 D_{ML;1}^{12.5;12.5} &= 0.0293 T^{0.5}, & D_{ML;1}^{18;18} &= 0.0296 T^{0.5}, \\
 D_{ML;1}^{22;28} &= 0.0300 T^{0.5}, & D_{ML;1}^{0;18} &= 0.0300 T^{0.5}, \\
 D_{ML;1}^{0;23} &= 0.0305 T^{0.5}, & D_{ML;1}^{10;30} &= 0.0310 T^{0.5}, \\
 D_{ML;1}^{0;40} &= 0.0335 T^{0.5}, & D_{ML;1}^{0;45} &= 0.0340 T^{0.5}
 \end{aligned} \tag{8}$$

for  $T \leq T_0$ , and

$$\begin{aligned}
 D_{ML;2}^{12.5;12.5} &= 0.1065 T^{0.9}, & D_{ML;2}^{18;18} &= 0.107 T^{0.9}, \\
 D_{ML;2}^{22;28} &= 0.1100 T^{0.9}, & D_{ML;2}^{0;18} &= 0.110 T^{0.9}, \\
 D_{ML;2}^{0;23} &= 0.1105 T^{0.9}, & D_{ML;2}^{10;30} &= 0.112 T^{0.9}, \\
 D_{ML;2}^{0;40} &= 0.1210 T^{0.9}, & D_{ML;2}^{0;45} &= 0.124 T^{0.9}
 \end{aligned} \tag{9}$$

for  $T \geq T_0$ , where  $D$  and  $T$  are measured in cm.

Quantitative results are gathered in Table I and compared with the data given in [5] for rectangular-prism-shaped samples of  $\varphi = 0$  orientation (respective edges parallel to the  $[110]$ ,  $[\bar{1}10]$  and  $[001]$  directions). For the sake of clarity, the table contains values only for some of the wedge-shaped samples, so selected that certain general regularities featuring the effect of studied factors on the  $D(T)$  dependence would be evident.

#### 4. Concluding remarks

The data obtained experimentally in this work provide further proof that in ferromagnetic substances the dependence of domain width on crystal thickness is described well by Eq. (1).

The performed experiments show that the angle of inclination of the wedge-shaped samples and the way in which they are cut (*i.e.* their crystallographic orientation) distinctly affect the  $D(T)$  curve. Table I and Fig. 4 show that (i) at a constant angle of inclination  $\vartheta + \theta$ , the  $D(T)$  curve lies the higher the more asymmetric is the wedge, *i.e.* the more the angles  $\theta$  and  $\vartheta$  differ, and (ii) that at a constant ratio of angles  $\vartheta$  and  $\theta$  the  $D(T)$  curve lies higher when the angle of inclination  $\vartheta + \theta$  is larger.

It also follows from the table and curves that even at a very large angle of inclination  $\vartheta + \theta$  the  $D(T)$  curve runs only slightly higher than the analogous curve for rectangular-prism-shaped samples of orientation  $\varphi = 0$ , if the wedge-shaped sample is symmetric crystallographically, *i.e.* if  $\vartheta = \theta$  (*cf.* columns 2 and 6 of Table I). As results from the data in columns 3, 4 and 6 of Table I, in the case of extremely asymmetric wedge-shaped samples ( $\vartheta = 0$ ) the results differ but slightly from those obtained from measurements on rectangular-prism-shaped samples of orientation  $\varphi = 0$  only when the angle of inclination is small. This explains the good agreement of the results of [4] and [5]. On the other hand, in the event of larger wedge angles the differences in domain widths at the sample crystal thicknesses are considerable (see columns 5 and 6 of Table I).

Hence, the implication of the performed investigations is that wedge-shaped samples of symmetric orientation ( $\vartheta = \theta$ ) are best suited for measuring the crystal thickness-dependence of domain width, at least for the *ML* domain structure.

We intend to show in our next paper that analogous studies made for the *SL* domain structure in FeSi lead to a similar conclusion. This principle probably also applies to uniaxial ferromagnetic substances, although this conjecture has not yet been verified in any experimental way. The conformity between the results of [1] and [2] for cobalt, even though a "one-sided" wedge-shaped sample was used, may most probably be explained by the small angle of inclination ( $18^\circ$ ) of the sample. On the other hand, there are considerable reservations regarding the results and conclusions (rather unprecise at that) of [11], where "one-sided" wedge-shaped samples of large angles of inclination were used.

The investigations presented here unfortunately still do not explain the causes for the appearance of a critical thickness  $T_0$  in three-axial ferromagnets. This fact is illustrated by the powder patterns reproduced in Figs 3b and c, of which the first shows the *ML* structure for  $T < 1.6 \times 10^{-2}$  cm  $< T_0$ , while the other for  $T > 1.75 \times 10^{-1}$  cm  $> T_0$ . For there are no evident essential differences in the geometry of the powder patterns at the sample surfaces, just as found in [4, 5].

The author is indebted to Dr W. J. Ziętek and Dr B. Wysocki for helpful scientific advice concerning this work.

#### REFERENCES

- [1] B. Wysocki, *Acta Phys. Polon.*, **34**, 327 (1968).
- [2] B. Wysocki, *Acta Phys. Polon.*, **35**, 179 (1969).
- [3] B. Wysocki, W. J. Ziętek, *Phys. Letters*, **29A**, 114 (1969).
- [4] B. Wysocki, *Acta Phys. Polon.*, **35**, 731 (1969).
- [5] S. Szymura, B. Wysocki, W. J. Ziętek, *Acta Phys. Polon.*, **A38**, 405 (1970).
- [6] G. Sołtysik, B. Wysocki, *Prace Inst. Hutniczych*, **19**, 247 (1967) (in Polish, English and Russian summary).
- [7] H. J. Williams, R. M. Bozorth, W. Shockely, *Phys. Rev.*, **75**, 155 (1949).
- [8] W. C. Elmore, *Phys. Rev.*, **51**, 982 (1937); **53**, 757 (1938).
- [9] J. R. Garrod, *Proc. Phys. Soc.*, **A79**, 1252 (1962).
- [10] B. Wysocki, W. J. Ziętek, *Postępy Fizyki*, **14**, 307 (1962), (in Polish).
- [11] Y. Takata, *J. Phys. Soc. Japan*, **18**, 87 (1963).