Zeitschrift für Geophysik, 1970, Band 36, Seite 113—124. Physica-Verlag, Würzburg

The Influence of the Dislocation Density and Inclusions on the Coercive Force of Multidomain Titanomagnetites of the Composition 0.65 $Fe_2TiO_4 \cdot$ 0.35 Fe_3O_4 in Basalts as Deduced from Domain Structure Observations

Von H. SOFFEL, München[1])

Eingegangen am 16. Juli 1969

Summary: The coercive force of multidomain homogeneous and almost spherical titanomagnetites (0.65 $Fe_2TiO_4 \cdot$ 0.35 Fe_3O_4) of two different basalts was studied using relationships for the interaction of domain walls with dislocation lines and nonferromagnetic inclusions. With the observed dislocation density ($5 \cdot 10^6/cm^2$) and the average number of inclusions in the titanomagnetites a maximal coercive force of the multidomain grains of about 100 Oe was evaluated. This is in good agreement with the demagnetization spectrum of the two basalts. Domain structure observations of the titanomagnetites in the basalts under the influence of external fields confirm the estimated figure for H_c.

Zusammenfassung: Die Koerzitivkraft annähernd kugelförmiger homogener Titanomagnetite (0.65 $Fe_2TiO_4 \cdot$ 0.35 Fe_3O_4) mit Mehrbereichskonfiguration in zwei verschiedenen Basalten wurde unter dem Gesichtspunkt der Wechselwirkung zwischen Blochwänden mit Versetzungslinien und unmagnetischen Einschlüssen untersucht. Mit der beobachteten Versetzungsdichte der Titanomagnetite ($5 \cdot 10^6/cm^2$) und der mittleren Anzahl der Einschlüsse ergab sich für die Mehrbereichsteilchen eine maximale Koerzitivkraft von etwa 100 Oe. Dieser Wert stimmt gut mit dem Entmagnetisierungs-Spektrum der beiden Basalte überein. Durch die Beobachtung der Bereichsstrukturen der untersuchten Titanomagnetite unter dem Einfluß äußerer Felder konnte der abgeschätzte Wert von H_c bestätigt werden.

Introduction

Independent of their chemical composition or structure pecularities the ferrimagnetic ore grains in the rocks can be classified into two categories with respect to the configuration of their magnetic domains, namely

(i) single domain particles,

(ii) multidomain particles.

In single domain particles the magnetization can only be changed by a rotation of the magnetization vectors from the directions of easy magnetization, which are determined by the crystal anisotropy, the shape anisotropy and eventual stress fields.

[1]) Privatdozent Dr. Heinrich SOFFEL, Institut für Angewandte Geophysik der Universität München, 8000 München 2, Richard-Wagner-Straße 10.

For an assembly of randomly oriented cubic single domain particles the coercive force H_c, i.e. the field which is necessary for a reversal of the magnetization vectors, is given by:

$$H_c = 0.64\, K/J_s \tag{1}$$

for pure crystal anisotropy,

$$H_c = 0.48\,(N_a - N_b)\, J_s \tag{2}$$

for pure shape anisotropy and

$$H_c = 1.44 \cdot \lambda_s \cdot \sigma / J_s \tag{3}$$

when an uniaxial stress σ is present. Hereby denote: J_s = saturation magnetization; K = crystal anisotropy constant; N_a and N_b the demagnetization factors; λ_s = magnetostriction constant. — The coercive force of single domain particles therefore mainly depends on parameters which are determined by the shape of the particles and by bulk magnetic properties, which are not sensitive for the real structure of the material.

In multidomain particles the changes of magnetization in weak magnetic fields (like the earth's magnetic field, $H_a < 0.6$ Oe) are produced by a motion of domain walls rather than by rotation processes. The mobility of the domain walls not only depends upon the bulk magnetic properties but also on parameters which are determined by the real structure of the crystal. Due to the real structure additional energy barriers are produced within the ore grains. The external field which is necessary to drive the domain walls across these energy barriers can be regarded as a kind of coercive force of a multidomain grain.

The theoretical considerations were tested by studying the mobility of the domain walls of multidomain titanomagnetite grains in basalts. The basalt samples are from Rauher Kulm and Parkstein, two tertiary basalts from the Oberpfalz, Germany. The rockmagnetic and paleomagnetic properties of these basalts were studied by REFAI [1961]; PETERSEN [1962] and SOFFEL [1968] for the Rauher Kulm basalt and by SOFFEL and SUPALAK [1968] and SOFFEL [1968] for the Parkstein basalt. The titanomagnetites of both basalts are homogeneous and the composition of both is $0.65\, Fe_2TiO_4 \cdot 0.35\, Fe_3O_4$.

The Influence of Inclusions and Lattice Imperfections on the Domain Wall Mobility in Ore Grains

Natural minerals always contain an appreciable amount of lattice imperfections which can be classified into: a) dislocations of different types, b) intersticial atoms. They also contain inclusions of nonmagnetic material, submicroscopic to microscopic in size. Due to the stress field and the magnetic stray field in their vicinity all these types of lattice imperfections and inclusions are able to interact with the corresponding fields around or within a domain wall.

For the interaction of a 180°-wall with a dislocation line only simplified models are available so far which have been developed for cubic and hexagonal metals [see SEEGER and coworkers, 1966]. There are good reasons to apply the same formula to the dense ferrimagnetic opaque minerals like magnetite, the titanomagnetites and hematite. According to TRÄUBLE [1966] 180°-walls can only interact with dislocation lines in the plane of the domain walls or which intersect them. Remote dislocation lines are assumed to have no effect on the mobility of a 180°-wall. This assumption however seems to be only a first order approximation according to recent investigations by MARKERT [1969].

Let us assume a dislocation line fixed to a knot and therefore unable to be shifted around easily in the material. The energy E_d per unit length of a dislocation line which is necessary to move a 180°-wall across the dislocation line is given after TRÄUBLE [1966, p. 267] to be:

$$E_d = \frac{3 \cdot G \cdot b \cdot \lambda \cdot \delta_w}{2} \sin\alpha \sin\omega . \tag{4}$$

Hereby denote: G = shear modulus; δ_w = wall thickness; b = burger's vector; λ = magnetostriction constant; ω and α the angles between the burger's vector and the normal of the domain wall or the dislocation line respectively. E_d has its maximal value for step-dislocations ($\alpha = 90°$) and b being normal to the domain wall ($\omega = 90°$). E_d is zero for screw dislocations ($\alpha = 0$) and/or b being parallel to the domain wall ($\omega = 0$).

The maximal force which a dislocation line of length l exerts on a 180°-wall is given by:

$$p_{\text{disl., max.}} = \frac{3}{2} G \cdot b \cdot \lambda \cdot l . \tag{5}$$

In the vicinity of a dislocation line the directions of magnetization deviate from the easy directions thus producing magnetic stray fields which are able to interact with the stray fields around a domain wall. For materials with large crystal anisotropy (for instance magnetite and possibly also the titanomagnetites) the deviations are very small and this stray field effect can be neglected besides the stress effect between a dislocation line and a domain wall as discussed above.

A much stronger interaction however must be expected between inclusions of non-ferromagnetic material and a domain wall. Such inclusions have been observed occasionally in the titanomagnetites of the investigated basalts. The interaction is threefold and is due to one or a combination of the following effects: (a) different stress state in the vicinity of the inclusion (E_σ), (b) reduction of the volume of a domain wall by the inclusion (E_{vol}), (c) the magnetic stray field around an inclusion (E_{str}). The total interaction energy between a domain wall and an inclusion is given by:

$$E = E_\sigma + E_{vol} + E_{str} \tag{6}$$

According to KRONMÜLLER [1962] E_σ can be neglected besides E_{vol} and E_{str}. The force p_{vol} which a spherical inclusion of a diameter $d \ll \delta_w$ exerts on a 180°-wall due to a reduction of the domain wall volume is given after DIJKSTRA and WERT [1950] to be:

$$p_{vol} = 2\pi d^3 \cdot \gamma_w / 3\,\delta_w^2 \tag{7}$$

γ_w denotes the specific energy and δ_w the thickness of a 180° domain wall. The force due to the stray field effect of small inclusions with $d \ll \delta_w$ is after the same authors given by:

$$p_{str} = \frac{2 \cdot \pi^4}{3} \left[\frac{1}{18} - \frac{1}{48} \right] \frac{J_s^2 \cdot d^5}{\delta_w^3} \tag{8}$$

Spherical inclusions occasionally have closure domains in their vicinity by which the stray field is considerably reduced. After KONDORSKY [1949] p_{str} is given in this case by:

$$p_{str} = 2\sqrt{2} \cdot \pi \cdot d \cdot \gamma_w . \tag{9}$$

The energy dE which is necessary for the movement dx of a 180°-wall of the area A by a magnetic field H is given by:

$$dE = 2 \cdot H \cdot J_s \cdot A \cdot \cos\varphi \cdot dx \tag{10}$$

whereby φ denotes the angle between the field H and the direction of J_s. Putting $\overline{\cos\varphi} = 2/3$, the force exerted by H on the domain wall is given by:

$$p = \frac{4}{3} H \cdot J_s \cdot A \tag{11}$$

and the field necessary to overcome an empeding force p is given by:

$$H = \frac{3 \cdot p}{4 \cdot J_s \cdot A} \tag{12}$$

This formula enables us to estimate the critical field strength (coercive force, H_c) which is necessary to move a 180°-wall across obstacles like pinned dislocation lines and inclusions. Combining (12) and (5) we yield the coercive force associated with a dislocation line:

$$H_{c,\,disl.} = \frac{9 \cdot G \cdot b \cdot \lambda \cdot l}{8 \cdot J_s \cdot A} \tag{13}$$

For an inclusion with $d \ll \delta_w$ we get with (12) and (7):

$$H_{c,\,\text{incl., vol}} = \frac{\pi \cdot d^3 \cdot \gamma_w}{2 \cdot \delta_w^2 \cdot J_s \cdot A} \tag{14}$$

and with (12) and (8):

$$H_{c,\,\text{incl., str}} = \frac{\pi^4}{2} \left[\frac{1}{18} - \frac{1}{48} \right] \frac{J_s \cdot d^5}{\delta_w^3 \cdot A} \tag{15}$$

For inclusions with $d \gtrsim \delta_w$ we yield with (12) and (9):

$$H_{c,\,\text{incl., str}} = \frac{3 \cdot \pi \cdot d \cdot \gamma_w}{2 \cdot J_s \cdot A} \tag{16}$$

Table 1 shows the coercive force for a magnetite and a titanomagnetite (0.65 Fe_2TiO_4 · 0.35 Fe_3O_4) grain of 10 by 10 by 10 microns by volume. For magnetite the specific energy of a 180°-wall and its thickness have been determined experimentally by SOFFEL [1964] to be $\gamma_w \approx 1$ erg/cm² and $\delta_w \approx 10^{-5}$ cm respectively. $\lambda_s = 40 \cdot 10^{-6}$ and $J_s = 480$ Gauss. The burger's vector is assumed to be equal to the unit cell dimension ($b \approx 8 \cdot 10^{-8}$ cm). The length l of a dislocation line cannot exeed the diameter of the grain (10^{-3} cm) and is certainly shorter (10^{-4} to 10^{-5} cm). The shear

Table 1: Coercive force of a multidomain ore grain, 10 by 10 by 10 microns by volume due to dislocation lines and spherical inclusions.

		Length l of a dislocation line (cm)				
		10^{-3}	10^{-4}	10^{-5}	10^{-6}	
$H_{c,\,\text{disl}}$ (Oe), after (13)	magnetite	1.5	0.15	0.015	0.0015	
	titanomagnetite	23	2.3	0.23	0.023	
		Diameter d of a spherical inclusion (cm)				
		$5 \cdot 10^{-4}$	10^{-4}	$5 \cdot 10^{-5}$	10^{-5}	10^{-6}
$H_{c,\,\text{vol}}$ (Oe), after (14)	magnetite	— model not applicable —			$3.3 \cdot 10^{-2}$	$3.3 \cdot 10^{-5}$
	titanomagnetite	— model not applicable —			$6 \cdot 10^{-3}$	$6 \cdot 10^{-6}$
$H_{c,\,\text{str}}$ (Oe), after (15)	magnetite	— model not applicable —			$8.1 \cdot 10^{-2}$	$8.1 \cdot 10^{-7}$
	titanomagnetite	— model not applicable —			$1.4 \cdot 10^{-3}$	$1.4 \cdot 10^{-8}$
$H_{c,\,\text{str}}$ (Oe), after (16)	magnetite	7	1.4	0.7	model not applicable	
	titanomagnetite	7	1.4	0.7	model not applicable	

modulus is $G = 2 \cdot 10^{11}$ dyn/cm^2 as computed after data from WOEBER, KATZ and AHRENS [1963].—For the investigated titanomagnetites much less information on some of the parameters are available. $J_s \approx 100$ Gauss according to their CURIE-temperatures and data from AKIMOTO, KATSURA and YOSHIDA [1957]. As magnetite and the titanomagnetites have essentially the same oxygen lattice both the shear modulus and the burger's vector are assumed to be the same as for magnetite. Measurements by SYONO [1965] of the magnetostriction constant λ_s and the crystal anisotropy constant K yielded the following values for the titanomagnetites under consideration: $\lambda_s = 140 \cdot 10^{-6}$ and $K = 10^4$ erg/cm^3. Both λ_s and K strongly influence the specific energy and thickness of domain walls, which is expressed by the following proportionalities:

$$\gamma_w \sim \sqrt{I_0 \cdot K} \tag{17}$$

and

$$\delta_w \sim \sqrt{I_0 / K} \tag{18}$$

[after KITTEL, 1949]. I_0 denotes the exchange integral, which is connected with the CURIE-temperatures by the relation:

$$T_c \sim I_0 \tag{19}$$

Equations (17) to (19) have been established so far only for metals with direct interactions between the magnetic moments of the atoms but not yet for the ferrimagnetic oxide minerals like magnetite or the titanomagnetites with superexchange interactions. It is assumed however that they can be applied to the ferrimagnetic oxides as well.

With the known values of γ_w and δ_w for magnetite the equations (17) to (19) enable us to compute the approximate values for the investigated titanomagnetites. For the exange integral we get therefore from (19):

$$\frac{I_{0,\,\text{titanomagn.}}}{I_{0,\,\text{magnetite}}} = \frac{460°\,\text{K}}{850°\,\text{K}} = 0.54 \tag{20}$$

As we have

$$\frac{K_{\text{titanomagn.}}}{K_{\text{magnetite}}} = \frac{10^4\,\text{erg/cm}^3}{10^5\,\text{erg/cm}^3} = 0.1 \tag{21}$$

we get for the specific wall energy from (17):

$$\gamma_{w,\,\text{titanomagn.}} = \gamma_{w,\,\text{magnetite}} \sqrt{\frac{(I_0 K)_{\text{titanomagn.}}}{(I_0 K)_{\text{magnetite}}}} \tag{22}$$

$$= \gamma_{w,\,\text{magnetite}} \sqrt{0.54 \cdot 0.1} = 0.23$$

With $\gamma_{w,\,\mathrm{magnetite}} \approx 1\ \mathrm{erg/cm^2}$ we get:

$$\gamma_{w,\,\mathrm{titanomagn.}} \approx 0.23\ \mathrm{erg/cm^2}\,. \tag{23}$$

The same estimation for the wall thickness yields the following result:

$$\begin{aligned}\delta_{w,\,\mathrm{titanomagn.}} &= \delta_{w,\,\mathrm{magnetite}}\sqrt{\frac{0.54}{0.1}} \\ &\approx 2.3\cdot 10^{-5}\ \mathrm{cm}\,.\end{aligned} \tag{24}$$

Table 1 shows that only dislocation lines of proper orientation with respect to the domain walls and of considerable length, and inclusions with a diameter larger or comparable to the thickness of a domain wall give remarcable contributions to the "wall friction" i.e. the coercive force of a multidomain grain. Intersticial atoms which can be regarded as inclusions with a diameter of about 10^{-7} cm have a negligable effect on the wall friction.

The total coercive force of a spherical multidomain grain therefore depends largely on the dislocation density, the length and orientation of the dislocation lines with respect to the domain walls and the special type of dislocation. It depends furthermore upon the abundance and size of nonferromagnetic inclusions. Another mechanism for the domain wall pinning resulting in a coercive force of a multidomain grain is due to deviations from the ideal spherical shape or by a very irregular shape of the ore grains. Both mechanisms do not apply to the investigated titanomagnetites and shall therefore not be discussed here.

Coercive Force of the Multidomain Grains as Deduced from the Observed Domain Wall Mobility in the Titanomagnetites

Due to the good spherical shape of most of the multidomain grains their coercive force is most likely exclusively due to the interaction of the domain walls with dislocation lines and inclusions of nonferromagnetic material with a diameter around 1 micron, according to table 1. The dislocation density was determined counting etch pits produced by ionic polishing as applied by Soffel [1968a] for the preparation of ferrimagnetic ore grains for domain structure observations. Fig. 1 shows a titanomagnetite grain in the Prakstein basalt with a diameter of about 8 microns (probably a 110-plane). The etch pits are interpreted as dislocation lines intersecting the surface. An average dislocation density of $5 \cdot 10^6/\mathrm{cm}^2$ was determined for the titanomagnetites of both the Parkstein and the Rauhe Kulm basalt. (A dislocation density of $5 \cdot 10^6/\mathrm{cm}^2$ means that an area of $10^{-6}\ \mathrm{cm}^2$, which is ten by ten microns, is intersected by 5 dislocation lines.) Fig. 2 shows the domain configuration of the ore grain as shown in Fig. 1 under the influence of external fields parallel to the polished surface of the grain.

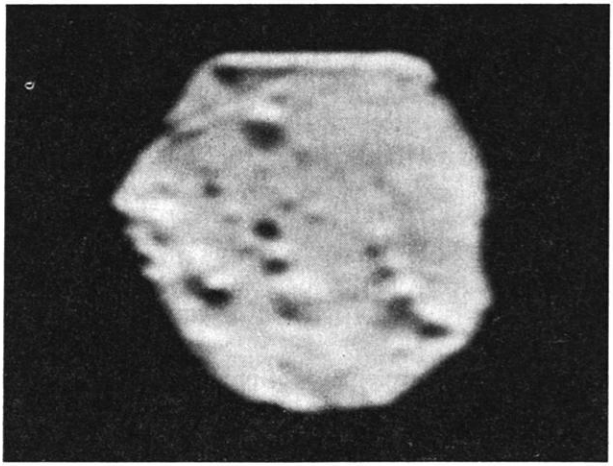

Fig. 1: Titanomagnetite grain in the Parkstein basalt, polished by ionic bombardment. The pits are dislocation lines intersecting the surface. For the scale see Fig. 3.

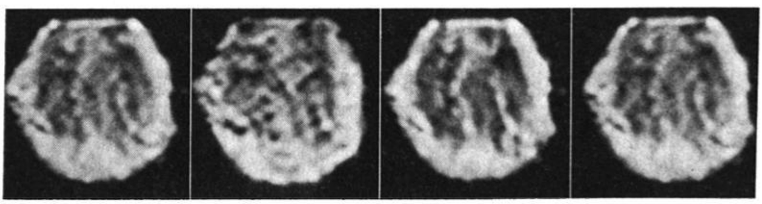

Fig. 2: Domain configuration of the ore grain as shown in Fig. 1 under the influence of external fields parallel to the surface (Bitter pattern technique). For the scale and the directions of the external fields see Fig. 3.

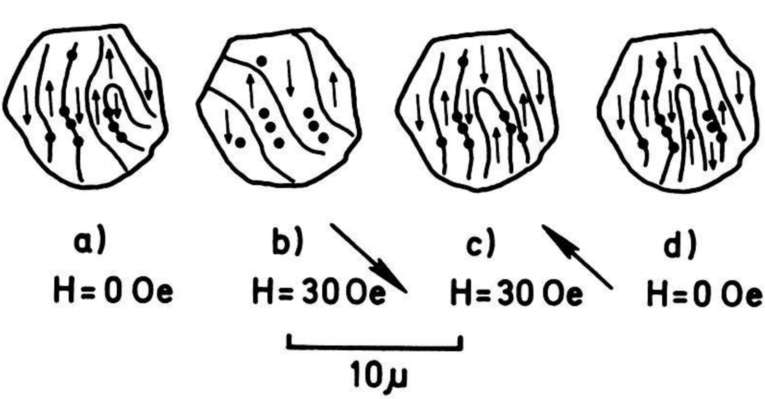

Fig. 3: Graph of the domain configurations as shown in Fig. 2. Dots: etch pits; bold lines: domain walls; small arrows: proposed magnetization directions of the domains; large arrows: direction of the external field.

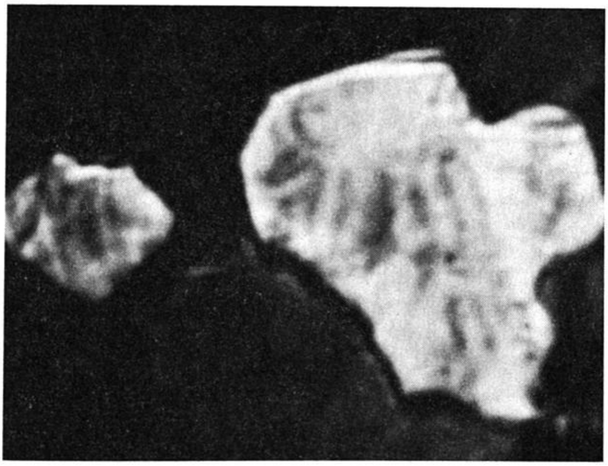

Fig. 4: Domain configuration of two titanomagnetite grains in the Parkstein basalt (Bitter pattern technique). For the scale see Fig. 5. Ambient field is zero.

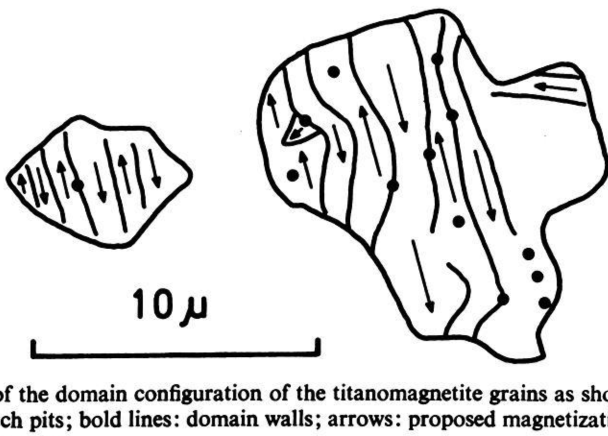

Fig. 5: Graph of the domain configuration of the titanomagnetite grains as shown in Fig. 4. Dots: etch pits; bold lines: domain walls; arrows: proposed magnetization directions of the domains.

The sample preparation was the same as described by Soffel [1968a]. The position of the etch pits (dots) and of the domain walls as deduced from photographs as well as from microscopic observations (bold lines) are shown in the graphs of Fig. 3. It can be seen that the domain walls are pinned to the etch pits (Fig. 3a, c, d) and that external fields in the order of 30 Oe are capable to separate them. The small arrows indicate a possible distribution of the directions of magnetization in the domains, as derived from the movement of the domain walls under the influence of the external field. Fig. 4 and 5 show another example of the same phenomenon for two other titanomagnetite grains in the Parkstein basalt.

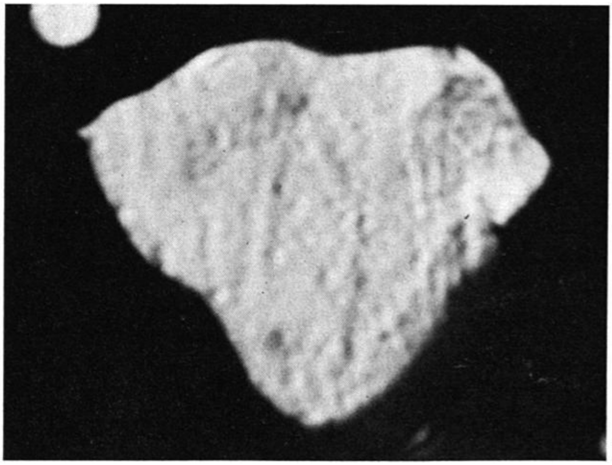

Fig. 6: Titanomagnetite grain in the Rauhe Kulm basalt, polished by ionic bombardment. The pits are dislocation lines intersecting the surface and inclusions. The thin straight lines are former scratches produced by previous mechanical polishing. For the scale see Fig. 8.

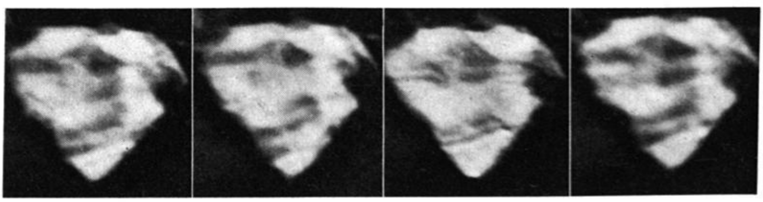

Fig. 7: Domain configuration of the ore grain as shown in Fig. 6 under the influence of external fields parallel to the surface (Bitter pattern technique). For the scale and the directions of the external fields see Fig. 8.

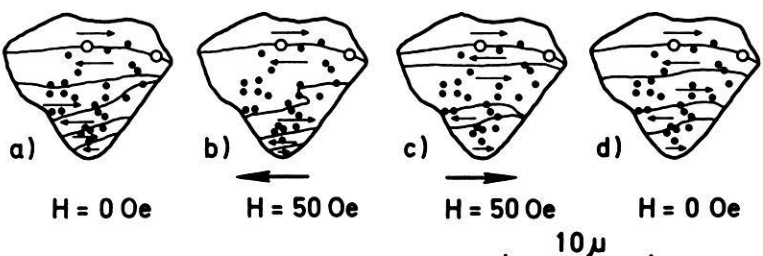

Fig. 8: Graph of the domain configuration as shown in Fig. 7. Dots: etch pits; open circles: inclusions; bold lines: domain walls; small arrows: proposed direction of magnetization of the domains; large arrows: direction of the external field.

Fig. 6 shows a titanomagnetite grain of about 10 microns in diameter in the Rauhe Kulm basalt. The variations of the domain configuration under the influence of external fields parallel to the polished surface of the grain is represented in Fig. 7. The inclusions (open circles), etch pits (dots), domain walls (bold lines) and magnetization directions in the domains (arrows) are shown in the graphs of Fig. 8. While domain walls pinned to dislocation lines can easily be moved under the influence of external fields in the order of 50 Oe, the two inclusions are able to hold the uppermost domain wall even in external fields higher than 50 Oe.

However only a minute fraction of the multidomain grains in both basalts contained such inclusions so that the dislocations are the major contributor to the coercive force of the multidomain grains of the investigated basalts.

Using the results listed in Table 1 and the actually observed dislocation density in the titanomagnetites ($5 \cdot 10^6/cm^2$), the coercive force of an average multidomain grain of the investigated titanomagnetites, say 10 by 10 by 10 microns by volume, is around 100 Oe or less. This figure is in good agreement with the demagnetization spectra of the two basalts which show a sharp drop at a demagnetizing field of about 100 Oe, as measured by SOFFEL [1969]. The value of 100 Oe can be regarded as an upper limit for the coercive force of the investigated titanomagnetites with multidomain configuration and is obtained when one assumes that all dislocations are step dislocations ($\alpha = 90°$) with the burger's vector normal to the wall ($\omega = 90°$) and that they have the length of the diameter of the crystal (10^{-3} cm). It is furthermore assumed that they interact with all domain walls, which must be expected according to the recent investigations by MARKERT [1969]. The results obtained from the domain structure observations agree fairly well with the above figures. The coercive force of multidomain grains increases considerably by additional interactions of inclusions with domain walls as shown in Fig. 8. The upper limit for the coercive force of a spherical ferrimagnetic ore grain due to intercations of the said lattice imperfections with the domain walls must be set between 100 Oe and 200 Oe. This is in agreement with the general picture of a demagnetization spectrum of an aggregate of multidomain grains in various rocks. Larger values for the coercive force of multidomain grains can occur for extremely high dislocation densities and a large number of inclusions in the ore grains (i.e. when the grain is subdivided by exsolution lamellae of a nonferromagnetic phase) or by elongated and/or very irregular shapes of the ore grains.

Acknowledgement

The investigations were carried out in the Institut für Angewandte Geophysik, Universität München. I am very indepted to its director, Prof. Dr. G. ANGENHEISTER for his support and to him and Dr. N. PETERSEN, Dr. A. SCHULT, Dr. E. SCHMIDBAUER, Dipl.-Geophys. J. POHL, Dipl.-Phys. U. BLEIL and Dipl.-Phys. H. MARKERT for their helpful discussions and suggestions. The financial support of the Deutsche Forschungsgemeinschaft is gratefully acknowledged.

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