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The domain structure of ferroelastic $BiVO_4$ studied by magnetic resonances

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Abstract. The domain structure of the ferroelastic $BiVO_4$ single crystal has been investigated using the nuclear magnetic resonance (NMR) of ^{51}V and by the electron paramagnetic resonance (EPR) of the Mn^{2+} ions contained in the crystal as an impurity. The 14 resonance lines of ^{51}V ($I = \frac{7}{2}$) in a $BiVO_4$ crystal with the twin domain were measured in the crystallographic a–b (or c–b) and c–a (or a–c) planes with an FT NMR spectrometer. Two sets of Mn^{2+} EPR signals were also obtained in the c–a plane. These two sets of NMR and EPR signals originate from the twin-domain structure. From these two sets of experimental data from ^{51}V NMR and Mn^{2+} EPR, it is confirmed that the $BiVO_4$ single crystal has the prominent (W-plane) domain wall reported previously. The investigated domain structure is found to be stable with time in contrast with a previous report by Baran *et al*. The previous model of the twinning mechanism derived from NMR and x-ray diffraction data has been improved by employing the EPR results. The observed W plane of the domain wall in $BiVO_4$ can be explained by the ferroelastic species $4/mmm\ F\ 2/m$ instead of $4/m\ F\ 2/m$.

1. Introduction

Ferroelastics have received considerable attention in recent years. $BiVO_4$ single crystals may prove to be a promising material for acousto-optics (Manolikas and Amelinckx 1980, Akimov *et al* 1982), and ferroelastic $BiVO_4$ crystal is a good material for studying the domain structure (Choh *et al* 1992). Since $BiVO_4$ was synthesized by Roth and Waring (1963), many studies (Pinczuk *et al* 1977, 1979, David *et al* 1979, Sleight *et al* 1979, Choh *et al* 1991, 1992) have been carried out. Ferroelasticity (Bierlein and Sleight 1975) and the second-order phase transition between the monoclinic fergusonite and tetragonal scheelite structures (Dudnik *et al* 1979) were found. The structural parameters for $BiVO_4$ are $a = 5.1966$ Å, $b = 11.704$ Å, $c = 5.0921$ Å and $\beta = 90.38°$ in the ferroelastic phase at room temperature (David *et al* 1979). Bierlein and Sleight (1975) suggested that $BiVO_4$ is the result of a ferroelastic transition of the Aizu (1970) type $4/m\ F\ 2/m$.

Dudnik *et al* (1975) studied the twin structure of $BiVO_4$ single crystals as a function of mechanical stress. Two sets of domain walls of $BiVO_4$ below 255 °C are oriented at $91.5 \pm 0.5°$ to each other, which compares favourably with the value of 91.14° calculated from the cell parameters (Bierlein and Sleight 1975). Another domain-wall direction from the a (or c) axis was obtained as $30 \pm 3°$ using a polarizing microscope (Sawada and

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Ishibashi 1979). The theoretically deduced angle between the boundaries and the a (or c) axis (31.4°) is in good agreement with the observed value (32°) (Manolikas and Amelinckx 1980). The calculated values between the W' walls and the [100] crystallographic direction gave an angle of 36°, in good agreement with the measured value (Wainer *et al* 1981). Mnushkina and Dudnik (1982) calculated an angle of 35° with the [100] direction. The relationship between the domain-wall orientation and the spontaneous strain in ferroelastics was discussed, with particular reference to the W' walls in $BiVO_4$, by David and Wood (1983). Four domains were observed with Gd^{3+} EPR in $BiVO_4$ after quenching the crystal from $T > T_c$ to $T = 300$ K (Baran *et al* 1985). However, the domain structure that they observed was metastable because all four domains disappeared as time passed (Baran *et al* 1985). Moon *et al* (1987) observed a new twin structure using x-ray diffraction and ^{51}V NMR: the domain boundary made an angle of 44.25° with respect to the a axis, different from the previous studies (Sawada and Ishibashi 1979, Manolikas and Amelinckx 1980, Wainer *et al* 1981). An angle of 44° for the twin-domain wall (W plane) can be obtained (Jeong *et al* 1987) using the ferroelastic theory proposed by Aizu (1970) and Sapriel (1975). Lim *et al* (1989) suggested that the W plane was also permissible taking the phase transition from $4/mmm$ to $2/m$ symmetry. Two kinds of nearly perpendicular domain wall were observed using a polarizing microscope, and the domain boundaries resulting from possible combinations of the four orientation states were discussed by Lim *et al* (1990). The domain wall of ferroelastic $BiVO_4$ studied by transmission electron microscopy also showed that the measured angle between the [100] axis and the domain boundaries is $44 \pm 1°$ (Lim *et al* 1992). All reported results for the W wall (prominent domain wall) and the W' wall (non-prominent domain wall) in a $BiVO_4$ single crystal are summarized in table 1.

Table 1. The observed and calculated values of the prominent and non-prominent domain-wall directions in a $BiVO_4$ crystal.

Reference	θ_{obs} (deg)	θ_{calc} (deg)	Domain wall	Experimental method
Sawada and Ishibashi (1979)	39		W'	Polarizing microscope
Manolikas and Amelinckx (1980)	32	31.4	W'	Electron microscopy; electron diffraction
Wainer *et al* (1981)	37.4	36	W'	TEM; electron diffraction
Mnushkina and Dudnik (1982)		35	W'	
		55		
David and Wood (1983)			W'	
Baran *et al* (1985)				Gd^{3+} EPR (metastable domain)
Moon *et al* (1987)	44.25		W	X-ray diffraction; NMR
Jeong *et al* (1987)		44	W	
Lim *et al* (1989)		45	W	
Lim *et al* (1992)	44		W	TEM; electron diffraction
Present work	44.2		W	^{51}V NMR; Mn^{2+} EPR (stable domain)

In our preceding papers, the domain structure was studied by x-ray diffraction and ^{51}V nuclear magnetic resonance (NMR) (Moon *et al* 1987), polarizing microscopy (Lim *et al* 1988, 1990), theoretical investigations (Jeong *et al* 1987, Lim *et al* 1989, Jang *et al* 1991) and transmission electron microscopy (Lim *et al* 1992). The Mn^{2+} electron paramagnetic resonance (EPR) in a $BiVO_4$ single crystal has been investigated experimentally and theoretically (Yeom *et al* 1992a, b, 1993). Although the twin-domain structure in a $BiVO_4$ crystal can be detected by means of x-ray diffraction, polarizing microscopy and NMR, an EPR analysis appears to be quite meaningful. The present work is an extension

of our study of the domain structure using the EPR of paramagnetic impurities in $BiVO_4$ because EPR is a sensitive technique for distinguishing domain structures (Baran *et al* 1985). Meanwhile, we have observed Mn^{2+} EPR centres in the ferroelastic phase of $BiVO_4$ single crystals with a single domain and with twin domains. In this paper, the domain structure is examined by means of the rotation patterns of the Mn^{2+} EPR spectra. Studies of x-ray diffraction and Fourier-transformed (FT) NMR data for ^{51}V in $BiVO_4$ are also presented for comparison with previous results (Choh *et al* 1985, Moon *et al* 1987).

2. Experimental aspects

$BiVO_4$ single crystals were grown by the Czochralski method with a mixture of Bi_2O_3 and V_2O_5 powder (Choh *et al* 1985). Single crystals with a single domain and with twin domains, confirmed by x-ray diffraction, were selected for NMR and EPR studies. The crystallographic principal axes of the specimen were determined by the x-ray Laue method. ^{51}V NMR spectra in $BiVO_4$ were obtained with a Bruker FT NMR spectrometer (MSL 200) operating at $\omega_0/2\pi = 52.640$ MHz. The stable magnetic field was 4.7 T. The NMR spectra of ^{51}V were recorded with a sequence of one 90°-pulse, four scans and a repetition time of 10 s. The pulse length was 1.1 μs, and a ring-down delay time of 7.7 μs was used to remove the effect of the pulse. The rotation patterns of the resonance fields on the crystallographic *a–b*, *b–c* and *c–a* planes were obtained at 5° intervals at room temperature.

The equipment used for the EPR measurements was a Bruker X-band EPR spectrometer (ESP 300 series) with a TE_{102} rectangular cavity. The microwave frequency during the measurements was kept in the range 9.800 ± 0.003 GHz for the single-domain crystal and 9.882 ± 0.003 GHz for the twin-domain crystal. The calibration of the magnetic field was accomplished with a Bruker NMR gaussmeter, while the microwave frequency was determined using the DDPH signal. The accuracy of the sample alignment was estimated to be ±0.5°. The rotation patterns of the resonance fields for Mn^{2+} EPR on the crystallographic *a–b*, *b–c* and *c–a* planes were obtained at 1–4° intervals at room temperature.

3. Theoretical background of magnetic resonance

The Hamiltonian for NMR to analyse the experimental results is the usual (Abragam 1961)

$$H = H_Z + H_Q \tag{1}$$

where H_Z is the Zeeman term and H_Q describes the nuclear electric quadrupole interaction of the ^{51}V ($I = \frac{7}{2}$) nucleus. The first-order perturbation of H_Q with respect to H_Z is considered in section 4. The frequency due to the first-order quadrupole interaction is

$$\nu_m^{(1)} = -\nu_Q \left(m - \tfrac{1}{2}\right)[(3\cos^2\theta - 1) + \eta \sin^2\theta \cos^2\varphi]$$

$$\nu_Q = 3e^2qQ/4I(2I-1)h \tag{2}$$

where m is the magnetic quantum number and η the asymmetry parameter. θ and φ are the polar and azimuthal angles, respectively, of the applied magnetic field B with respect to the principal axes of the electric field gradient (EFG) tensor.

The experimental results from Mn^{2+} EPR in a $BiVO_4$ single crystal can be analysed with the usual spin Hamiltonian (Abragam and Bleaney 1970, Rudowicz 1987)

$$H = H_Z + H_{zfs} + H_h \tag{3}$$

where the terms on the right-hand side are the Zeeman, the fine structure (zero-field splitting) and the hyperfine interactions, respectively.

4. Experimental data and analysis

The single- and twin-domain structures of $BiVO_4$ crystals were pre-examined with the x-ray diffraction method for comparison with our previous study (Moon *et al* 1987). The rotation patterns of the ^{51}V NMR spectra were measured with the single- and twin-domain crystals on the crystallographic *a–b*, *b–c* and *c–a* planes. While seven resonance lines of the ^{51}V nucleus ($I = \frac{7}{2}$) observed in the single-domain $BiVO_4$, two sets of seven lines were recorded in the twin-domain crystal. A typical NMR spectrum of ^{51}V measured with the pulse NMR spectrometer is shown in figure 1. It is a Fourier transform of the free-induction decay for the ^{51}V NMR. The central transition is stronger than the satellite lines, and the separations between adjacent lines are almost equal. The zero point of the *X* axis in figure 1 corresponds to the resonance frequency 52.640 MHz of the bare ^{51}V nucleus. These experimental results are in good agreement with the observation using a continuous-wave NMR spectrometer (Moon *et al* 1987).

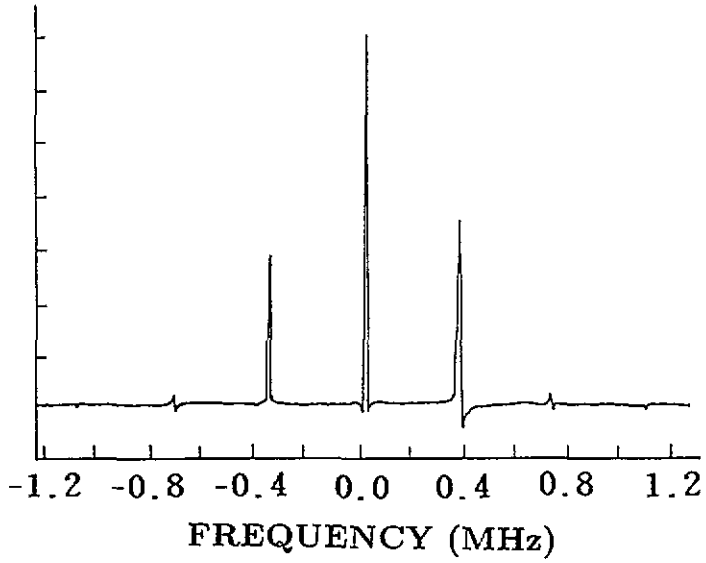

Figure 1. A typical NMR spectrum of ^{51}V in a $BiVO_4$ crystal recorded with the pulse NMR spectrometer. The static magnetic field B_0 is parallel to the *b* axis. The numbers on the horizontal axis are the frequency differences from the reference frequency (52.640 MHz).

The rotation pattern in the *c–a* (*a–c*) plane with a twin-domain crystal is shown in figure 2. The nuclear quadrupole coupling constant e^2qQ/h and asymmetry parameter η were determined from equation (2) using the NMR spectra measured in the crystals with the single domain and the twin domains, respectively. The first-order perturbation of H_Q with respect to H_Z was sufficient because the spacings between adjacent resonance lines were almost equal. The values of $e^2qQ/h = 4.88 \pm 0.03$ MHz and $\eta = 0.38 \pm 0.01$ obtained here are in good agreement with previous values (Choh *et al* 1985) within the experimental accuracy. It turns out that figure 2 is a superposition of two rotation patterns, one in the *a–c* and one in the *c–a* plane. Rotation patterns in the *a–b* and *c–b* planes are also superposed. This shows that the crystallographic *a* and *c* axes of one domain are rotated by an angle of 90° to become the crystallographic *a* and *c* axes of the other domain. In particular, when

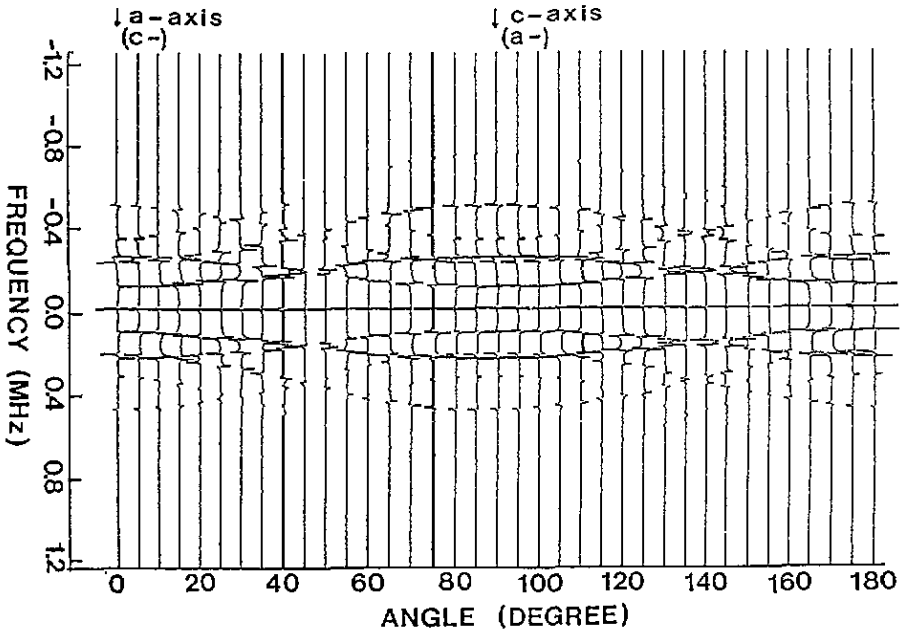

Figure 2. Rotation pattern of the pulse NMR spectra of ^{51}V in a twin-domain $BiVO_4$ crystal measured in the c–a (and a–c) plane at 300 K.

an external magnetic field is applied along the b axis, the two sets of resonance lines of the twin-domain crystal coincide with that of the single-domain crystal as shown in figure 1.

Two sets of the EPR spectrum for Mn^{2+} in the twin-domain crystal measured at room temperature are shown in figure 3, where we deal only with the spectrum of the Mn^{2+} ion. The ten groups of lines labelled 1′–5′ as well as 1–5 are the fine structure of $Mn^{2+}(S = \frac{5}{2})$, and the six lines within each group are the hyperfine structure of ^{55}Mn ($I = \frac{5}{2}$; 100% natural abundance). The five groups of lines labelled 1–5 correspond to those of the fine structure when the magnetic field is applied along the Z axis of the tensor **D** in the crystallographic a–c plane for a single crystal with a single domain, whereas the the other five groups labelled 1′–5′ correspond to those when the static magnetic field is parallel to the Y axis of **D** in the a–c plane of the single-domain crystal. The signal intensity of one set (the primed) is stronger than that of the other set (unprimed). This shows that there are two different domains having different sizes. The rotation pattern of Mn^{2+} EPR in the a–c (and c–$\bar{a}$) plane of the twin-domain crystal is shown in figure 4. The two domains are magnetically inequivalent when the static magnetic field is in the a–c plane. The rotation patterns in figure 4 display simultaneously one Mn^{2+} centre and another displaced by 90°. The rotation patterns in the a–c plane (full circles) and the c–$\bar{a}$ plane (open circles) for the single-domain crystal are superposed in the twin-domain crystal. The domain structure that we obtained from ^{51}V NMR and Mn^{2+} EPR turn out to be stable, unlike that observed with Gd^{3+} EPR (Baran *et al* 1985).

5. Results and discussion

The principal axes of the EFG tensor for ^{51}V in the single-domain crystal show that X', Y'

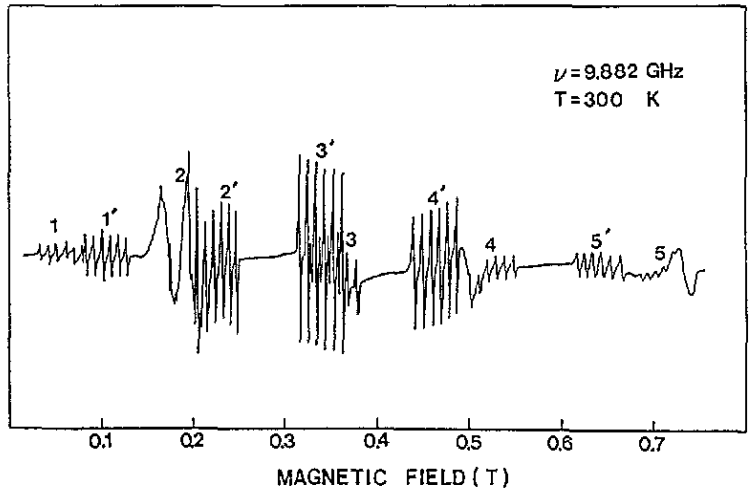

Figure 3. EPR spectra of Mn^{2+} and other impurities in a $BiVO_4$ single crystal with a twin domain. The magnetic field was applied along the Z (and Y) axis of the **D**.

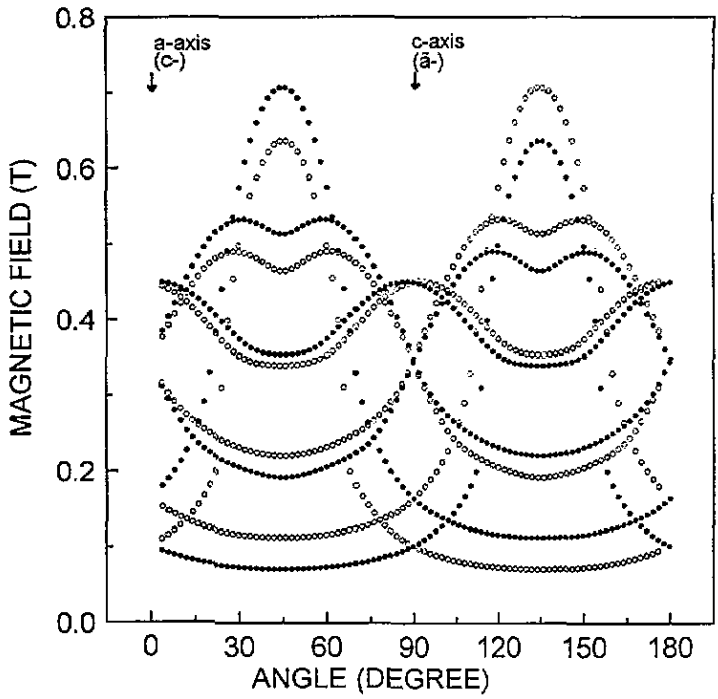

Figure 4. Rotation pattern for the Mn^{2+} EPR measured at 300 K in the a–c plane of one (●) domain of the twin-domain crystal; ○, pattern originating from the other domain.

and Z' are parallel to the crystallographic c, a and b axes, respectively, in agreement with a previous study (Moon *et al* 1987). From the ^{51}V NMR study, the twin structure of $BiVO_4$

is shown in figure 5(*a*) and is as adopted previously (Moon *et al* 1987). This indicates that the mirror plane of the twin domains is the bisector of the *a*–*c* plane, where the angle β ($= 90.38°$) is only slightly different from 90°.

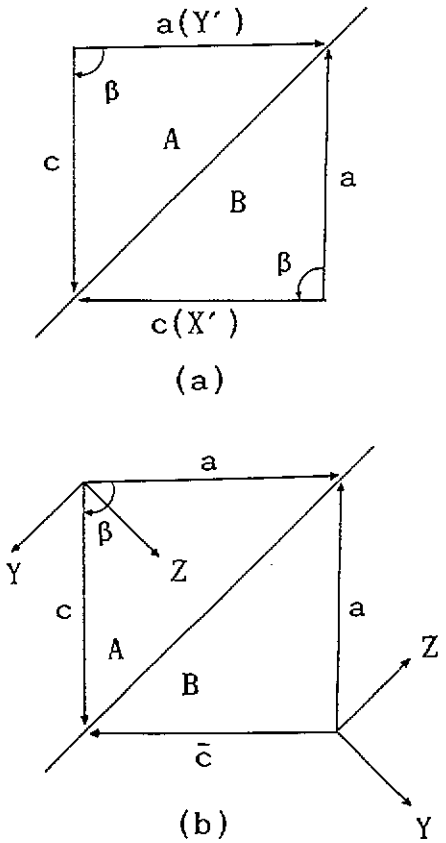

Figure 5. The twinning structure of $BiVO_4$ ($\beta = 90.38°$). (*a*) The twinning mechanism obtained with ^{51}V NMR. The X', Y' and Z' axes are the principal axes of the EFG tensor. (*b*) The twinning mechanism derived from Mn^{2+} EPR. The X, Y and Z axes are the principal axes of **D**.

The principal X, Y and Z axes of the tensor **D** for Mn^{2+} in single-domain $BiVO_4$ are found to be along the crystallographic $\bar{b}$, $c + 45°$ and $\bar{a} + 45°$ axes, respectively (Yeom *et al* 1992a). Using the spin Hamiltonian (equation (3)) the two sets of EPR parameters for Mn^{2+} in the twin-domain $BiVO_4$ are found to be the same except for the 90° discrepancy of the principal axes of **D**. The EPR parameters obtained in the twin-domain crystal agree with those of the single-domain crystal (Yeom *et al* 1992a). The Mn^{2+} EPR lines in the twin-domain crystal, shown in figure 4, are a superposition of the resonance lines of one domain and lines rotated by an angle of 90° in the single-domain crystal. From these results, one can deduce that the $BiVO_4$ single crystal possesses two domains, having the *a* axis in one domain parallel to the *c* axis in the other domain. Because the principal Y and Z axes of **D** for Mn^{2+} are along the crystallographic $c + 45°$ and $\bar{a} + 45°$ axes, respectively, the domain structure shown in figure 5(*a*) cannot explain the two-group signals of the Mn^{2+} ion in figure 4. A NMR study of ^{51}V in $BiVO_4$ cannot differentiate between the *c* and $\bar{c}$

axes nor between the a and $\bar{a}$ axes because of the parallel relation between the principal axes of the EFG tensor and the crystallographic axes ($X' \parallel c, Y' \parallel a$ and $Z' \parallel b$). From the EPR study of the Mn^{2+} ion in the twin-domain crystal, the twinning structure shown in figure 5(a), adopted from the ^{51}V NMR study, had to be modified as shown in figure 5(b). The principal axes of the **D** for the Mn^{2+} ion are related to the crystallographic axes, as shown in figure 5(b). The boundary between the twin domains can be described by the integer Miller indices (the prominent W-domain wall) in the approximately orthorhombic structure of $BiVO_4$. Therefore, it can be concluded that there is a W wall, as shown in figure 5(b), in the twin-domain crystal. This result is consistent with our previous studies (Choh *et al* 1985, Moon *et al* 1987, Lim *et al* 1988, 1989, 1990, 1992).

Sapriel (1975) stated that the tetragonal system is tetragonal 1 ($4/mmm$) and 2 ($4/m$) in the 11 Laue classifications. There are three kinds of domain-wall orientation in a ferroelastic material having monoclinic symmetry and transforming from tetragonal 1 ($4/mmm$) to the monoclinic ($2/m$). Using one of the three domain-wall orientations, Jang *et al* (1991) calculated that an angle of 44.5° for the W wall is possible and that the W' wall (non-prominent domain wall) exists in the case of the phase transition from the tetragonal 2 ($4/m$) to the monoclinic ($2/m$) as well. The possible domain walls between two domains for the ferroelastic material are two W planes, one W plane and one W' plane, and two W' planes (Sapriel 1975). According to our previous reports obtained using polarizing microscopy (Lim *et al* 1990, Choh *et al* 1991), two kinds of domain state exist, namely a W wall as well as two W walls which are nearly perpendicular to each other in the $BiVO_4$ single crystal having a twin-domain structure. However, according to the present study of the Mn^{2+} EPR, the domain structure of the two W walls in our previous study (Lim *et al* 1990) should be modified as shown in figure 6.

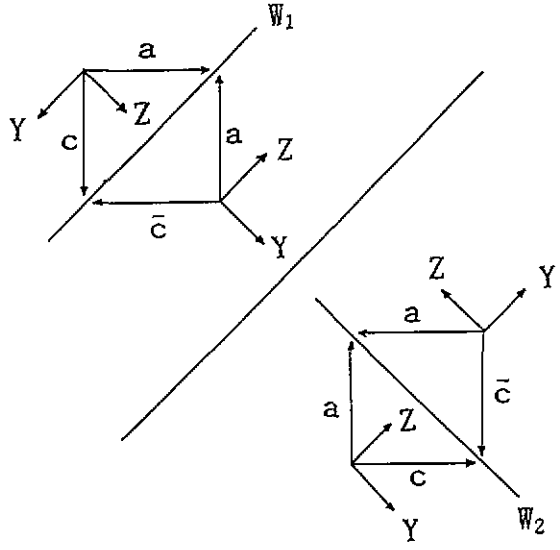

Figure 6. The twinning mechanism for the twin structure of $BiVO_4$ with two W planes. The X, Y and Z axes are the principal axes of **D**.

The W' domain wall of the $BiVO_4$ crystal has been reported in many other studies as well. The formation of single- or twin-domain crystals seems to be dependent on

the conditions of crystal growth. Natural $BiVO_4$ single crystals, occurring as the mineral pucherite, are known to have an orthorhombic structure. Apparently, laboratory syntheses have never achieved this modification. Low-temperature syntheses produce a tetragonal zircon-type $BiVO_4$ and high-temperature syntheses result in a monoclinic form of $BiVO_4$ (Bierlein and Sleight 1975). Consequently, $BiVO_4$ single crystals may have different crystal structures according to the conditions of the natural and laboratory syntheses.

From the above discussions, the twin $BiVO_4$ crystals that we employed in this and previous studies have a symmetry allowing the phase transition from tetragonal 1 ($4/mmm$) to monoclinic ($2/m$) having only one or two prominent domain walls.

6. Conclusion

The domain structure of the ferroelastic $BiVO_4$ crystals has been studied by ^{51}V FT NMR and Mn^{2+} EPR. All twin-domain crystals grown by our group have only prominent domain walls (one or two W planes) without any non-prominent domain walls. Thus, we may conclude that our crystals have a symmetry allowing the phase transition from $4/mmm$ to $2/m$. The domain structure in our crystals obtained from x-ray diffraction, ^{51}V NMR and Mn^{2+} EPR turns out to be stable instead of metastable as in a previous report. A model of the twin structure for the ferroelastic $BiVO_4$ crystal is newly suggested from the Mn^{2+} EPR study. This model can also explain the previous x-ray diffraction, polarizing microscopy and NMR experimental results.

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