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Climb dissociation in \{113\} planes in Al-Mg spinels

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Abstract. — Dislocations in Mg-Al spinels exhibit a very peculiar phenomenon. At high temperature they are dissociated off their glide plane (climb dissociation). \{100\} and \{110\} dissociation planes have already been characterized. One reports here experimental evidence that \{113\} planes are also possible dissociation planes with similar fault energies. Comparison of these various possible faults is made on the basis of crystallographic considerations (mismatches in the cationic configurations in and around the fault planes). It is found that, at least for non equimolar spinel, because of the large cationic vacancy density, \{113\} faults should not be more energetic that the other ones, as far as these faults are electrically neutral (i.e. the removed layer does not change the chemical composition of the material).

1. Introduction. — High temperature plasticity of Mg-Al spinels i.e. MgO.nAl_{2}O_{3} with 1 ≤ n ≤ 3.5 has been extensively studied in the recent past (see reviews in [1-3]). An important and interesting phenomenon has been now clearly established in this material : at high temperature, non screw dislocations are dissociated in planes which do not contain the Burgers vectors of the partials [4-7]. Such dissociations can only be achieved by climb, but once dissociated, the glide motion of the total dislocation implies continuous rearrangement of cations only, because the anionic sublattice is not faulted by the partials \(a/4\) \(< 110\). This cationic diffusion should be strongly dependent on the density of structural cationic vacancies \(V_{c} = (n - 1)/3(3n + 1)\). Deformation tests effectively show a much higher elastic limit for equimolar spinels [2, 5, 8-10]. Detailed mechanisms for this diffusion controlled (or viscous) glide for various \(n\) are discussed in another paper [11].

In the present article we report new experiments, especially T.E.M. investigations, which show that dislocations can also be dissociated by climb in \{113\} planes as far as the corresponding non conservative faults do not affect the chemical composition \(n\), i.e., the dislocations are not electrically charged. We then compare the various non conservative faults already characterized in spinels i.e. \(a/4\) \(< 01\overline{1}\) as displacement vector (partial dislocation Burgers vector) in fault planes \{011\}, \{001\} and \{113\} ; the first plane corresponds to a pure climb dissociation while in the two other cases the Burgers vector makes an angle of 45° and 32° respectively with the normal to the dissociation plane. In the absence of complete calculations of fault energies, our comparisons are based on simple crystallographic considerations i.e. on cationic arrangements (intercation distances) in and around the fault planes for various compositions. It is found that for \(n > 1.8\), faults in \{113\} lead to a cationic mismatch density very similar to the one found in \(a/4\) \{011\} \{011\} which is generally considered as the least energetic fault in the spinel structure.

(*) Associated to C.N.R.S. n° 234.
These qualitative arguments fully agree with our observations in 1.8 spinel and with the ones already published by Donlon et al. [7] for 3.5 spinel.

2. New experimental results. — Three types of T.E.M. observations have confirmed the occurrence of faults in $\{113\}$ planes in 1.8 spinels: i) observation of as grown extended faults; ii) preferred orientations of dislocations after a slight deformation at moderate temperature and iii) direct characterization by standard stereographic methods of the fault plane of dissociated dislocations imaged with the weak-beam technique [6].

2.1 As grown extended faults. — They have been observed in a non deformed crystal (1) with a molar ratio $n = 1.8$. As clearly shown on figure 1 these faults zig-zag in two different planes. A complete and detailed characterization leads to the schematic representation on figure 1e. The fault planes are $(100)$ and $(311)$ while the displacement vectors are $R_1 = a/4[101]$ for the upper part and $R_2 = a/4[110]$ for the lower part. The faults end on partial dislocations with Burgers vectors $b_1 = R_1$ and $b_2 = R_2$ at the ends, while in the middle the partial Burgers vector is $b_3 = a/4[111] = b_1 + b_2$. From a purely geometrical point of view this configuration is identical to the one which would result from the dissociation of two perfect dislocations with Burgers vectors $a/2[101]$ and $a/2[110]$ for the upper and lower parts respectively and with an attractive junction in the central region. However the dissociation surfaces oscillate between $(100)$ and $(311)$ forming several steps (see Fig. 1) with a mean length of 1 700 Å; this value is much larger than the dissociation width observed in $n = 1.8$ deformed spinel (order 200 Å). The configuration shown on figure 1 is thus probably out of equilibrium. However it has to be kept in mind that the equilibrium dissociation width seems to increase with increasing $n$ and increasing $T$ (for instance Donlon et al. [7] observe dissociation width $\sim 650$ Å in $n = 3.5$ spinel deformed at $T = 1 500$ °C). The observed feature on figure 1 might thus be due either to a large local deviation of the molar ratio $n$ or it might be a relictual equilibrium configuration formed at very high temperature during the growth process.

Whatever the exact origin of these defects, they show that non conservative faults in $\{113\}$ as well as in $\{100\}$ do exist in non equimolar spinels and are not destroyed by a short time annealing ($\sim 1$ hour at $1 400$ °C).

2.2 Preferred orientations. — It has already been pointed out that $a/2 \langle 110 \rangle \{110\}$ are the easy glide systems for $n = 1.8$ spinel [8, 9, 15]; for small deformations ($< 1 \%$ at $T = 1 400$ °C) one observes very long and straight dislocations lying along low index crystallographic directions. Figure 2 illustrates this point. In the $(011)$ thin foil, dislocations with Burgers vector $a/2[01\bar{1}]$ lie along $(100)$, $(411)$ and $(411)$ directions. The former one corresponds to the edge orientation, at the intersection with $(011)$ which has been shown to be the climb dissociation plane. It should be noted that many of these pure edge dislocations are in fact dipoles or dipole arms. They still are visible at large deformations and they could account for the vague tendency to edge orientation mentioned in our earlier paper [15]. In contrast $\langle 114 \rangle$ orientations disappear after 1 or 2 % strain.

For these $\langle 114 \rangle$ preferred orientations there are a lot of possible climb dissociation planes with relatively low indices. For instance, the following planes intersect $(011)$ along $(411)$: $(413)$; $(131)$; $(115)$; $(151)$; $(104)$; $(140)$; $(122)$... However the correct fault plane should also be evidenced in $\{111\}$ glide by one of the marked preferred orientations: screw, edge and $\pm 60^\circ$ character [6]. This comparison eliminates all the planes but the $\{113\}$ and the $\{115\}$ ones. Furthermore, as shown below, we find in other samples, by direct stereographic characterization, dissociation in $\{113\}$ but not in $\{115\}$.

All these arguments strongly suggest that the dislocations shown on figure 2, lying along $(411)$ and $(411)$ are dissociated in $(113)$ or $(131)$ and $(113)$ or $(131)$ respectively.

2.3 Weak beam characterization of dissociation in $\{113\}$. — The stereographic analysis of the dissociation plane has been performed in a sample with a thermomechanical history very similar to the ones of Donlon et al. [7]. This 1.8 spinel sample has been first slightly deformed at $T = 1 400$ °C, then it has been heated at $1 600$ °C and reloaded for a very short time. The resulting dislocations appear much more widely dissociated (up to 500 Å, while in usual tests with the same molar ratio, dissociation widths do not exceed 200 Å at similar temperature). The reason for such a widening is not completely elucidated at the moment. It could be due to a local variation of the molar composition on the climbing dislocation cores [16].

Whatever the exact mechanism for the ribbon widening, the stereographic determination of the fault planes is rendered much easier by this thermal treatment. The stereographic technique has already been described [6]. In brief, it consists in comparing the variation of the apparent dissociation width of a given
Fig. 1. — (111) foil of $n = 1.8$ spinel. Two large non conservative faults zig zagging between the (100) and the (311) fault planes. The displacement vectors are $a/4 [101]$ for the upper fault and $a/4 [110]$ for the lower one. a), b), c), d) are T.E.M. micrographs with different diffraction conditions imaging either the faults or the partial dislocations. e) schematic representation of the faults.
Fig. 2. — Specimen \( n = 1.8 \) very slightly deformed at \( 1,400 \, ^\circ\text{C} \) (\( \varepsilon < 1 \% \)). The thin foil plane (011) is parallel to the glide plane, \( g = 044 \). The dislocations exhibit preferred orientations parallel to [411] and [411]; these dislocations are dissociated in \{111\} planes.

Dislocation, imaged in weak-beam technique, for various tilt angles, with the predicted variation for various postulated fault planes in zone with the dislocation line. The best fit is then considered as the dissociation plane. Of course an arbitrary plane \((hkil)\) can only be found as dissociation plane if it has been postulated (this rather primitive method could certainly be considerably improved in computerizing it; this would allow any plane in zone with the dislocation to be tested).

Our weak-beam observations have been performed on (100) foils which allow good weak-beam conditions to be reached. We find long dislocation segments slightly inclined to the (100) foil, like the one shown on figure 3, with a Burgers vector \( b = a/2 \, [101] \). Their orientation slightly changes when their dissociation plane changes from (311) on the right side to (100) on the left side. This change occurs without constriction (the displacement vector \( R = a/4 \, [101] \) is the same for both faults). The thermal treatment

Fig. 3. — Specimen \( n = 1.8 \) slightly deformed at \( 1,400 \, ^\circ\text{C} \), then reloaded for a short time at \( 1,600 \, ^\circ\text{C} \). (100) thin foil; weak-beam conditions with \( g = 044, s_\alpha = 2 \times 10^{-2} \, \text{Å}^{-1}, w = |s_\alpha x y| \sim 17 \). a) the thin foil is tilted around the \( X \) axis : \( \tau = - 6^\circ \); b) the thin foil is tilted around the \( X \) axis : \( \tau = + 28^\circ \). The Burgers vector of this dislocation is \( b = a/2 \, [101] \); its dissociation plane changes along the ribbon, without constriction : it is (311) on the right side and (100) on the left one.
suffered by the sample enlarges the dissociation widths (300 Å and 540 Å in (311) and (100) respectively) which are probably out of equilibrium in our experiment; this could be the case for Donlon et al. observations too [7]. However these results show that {100} and {113} fault planes have similar low fault energy densities. Both types of sessile-climb-dissociations probably occur in \( n = 1.8 \) deformed spinel.

3. Discussion. — The spinel structure appears to have a wide variety of climb dissociation configurations leading to numerous preferred dislocation orientations in samples slightly deformed by glide. There is an important question: does the molar ratio \( n \) influence the choice of the dissociation plane? In almost equimolar spinel \( n = 1.1 \), we have found that the 60° segments of dislocation loops gliding in {111} are dissociated in {100} [6]; but as pointed out above, with our primitive method used for the stereographic characterization of the dissociation plane, the possible

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Fig. 4. — Determination of the dissociation plane for a 60° dislocation in \( n = 1.1 \) spinel: a) experimental curve \( \delta \) (apparent ribbon width) versus \( \varphi \) (tilt angle of the thin foil in the microscope); b) curves \( \delta/d \) versus \( \varphi \) (\( d \) is the dissociation width) calculated for various possible dissociation planes; solid lines: our previous hypotheses [6]; broken lines: the new hypotheses (113) and (113); c) the experimental data fit very well the calculated curve for (113); d) schematic representation of the various planes in zone with the direction of the dissociated dislocation.
Table I. 

Cation mismatches for non conservative faults. (T and O represent cations in tetrahedral and octahedral sites respectively.)

<table>
<thead>
<tr>
<th>Fault</th>
<th>Shortest $T-O$ distance</th>
<th>Number of $T-O$ mismatches per $a^2$ faulted surface</th>
<th>Shortest $T-T$ distance</th>
<th>Number of $T-T$ mismatches per $a^2$ faulted surface</th>
</tr>
</thead>
<tbody>
<tr>
<td>Perfect crystal</td>
<td>—</td>
<td>—</td>
<td>0.434 $a$</td>
<td>0</td>
</tr>
<tr>
<td>$a/4 [01\overline{1}] (01\overline{1})$</td>
<td>0.414 $a$</td>
<td>0</td>
<td>0.354 $a$</td>
<td>2.8</td>
</tr>
<tr>
<td>$a/4 [01\overline{1}] (001)$</td>
<td>0.217 $a$</td>
<td>2</td>
<td>0.354 $a$</td>
<td>2</td>
</tr>
<tr>
<td>$a/4 [0\overline{1}\overline{1}] (11\overline{3})$</td>
<td>0.217 $a$</td>
<td>1.2</td>
<td>0.354 $a$</td>
<td>2.4</td>
</tr>
</tbody>
</table>

Fault planes have to be postulated, then they are compared to the experimental results. As we had not considered the $\{ 113 \}$ planes in zone with the $60^\circ$ dislocations in $[6]$, we could not find them. We have thus reconsidered in detail our previous experimental data on $n = 1.1$ spinel. We find that, at least in some cases, $\{ 113 \}$ planes $^{(2)}$ lead to a much better fit (Fig. 4). Therefore we have no serious experimental argument for the possible influence of $n$ on the relative occurrence of the various climb dissociations.

Actually we have fully characterized only three non conservative faults namely :

- $a/4 [01\overline{1}] (01\overline{1})$ associated to the edge dislocations in $\{ 110 \}$ and $\{ 111 \}$ glides.
- $a/4 [01\overline{1}] (001)$ associated to the $\pm 60^\circ$ dislocations in $\{ 111 \}$ glide.
- $a/4 [0\overline{1}\overline{1}] (11\overline{3})$ associated to the $\pm 60^\circ$ dislocations in $\{ 111 \}$ glide and to dislocations with $70^\circ$ character in $\{ 110 \}$ glide ($\langle 114 \rangle$ directions).

Note that the fault vector $R = a/4 [0\overline{1}\overline{1}]$ is a lattice period of the anionic sublattice which is not affected by any of these faults. Moreover, all these faults are non conservative ($R$ does not lie in the fault plane), but all are neutral and keep unchanged the composition (i.e. the removed layer has the nominal composition $\text{MgA}^2\text{O}_4$, see Appendix). This is not the case for most of the faults characterized by Donlon et al. $[7]$ in a $n = 3.5$ spinel deformed at $1500^\circ\text{C}$. In this $3.5$ spinel with a large cation vacancy density, segregation of vacancies on the fault plane could balance the charge and in that case only, $n$ would influence the choice of the dissociation planes.

The fault energies of the two first faults have been calculated by Veyssière et al. $[13, 14]$, but the energy of the third one is not yet known (calculations are in progress). One can thus only compare these faults from a crystallographic point of view in considering the intercation distances in and around the fault plane. Such an approach has already been used by Van der Biest and Thomas $[12]$. Table I gives the density of various cationic mismatches for these three faults in $n = 1$ spinel while the faults are schematically represented on figure 5.

Assuming that the major contribution to the fault energy comes from adjacent cation-cation interactions (i.e. one neglects long range interactions, and the anionic sublattice is not affected by the fault), it is seen from table I that the pure climb dissociation fault $a/4 [01\overline{1}] (01\overline{1})$ corresponds to the minimum density of mismatches. It should thus lead to the largest dissociation. This is effectively observed in $1.1$ spinel: we find $[6]$ $d(01\overline{1}) = 100 \text{ Å}$ for the edge dislocations while the $60^\circ$ ones give either $d(001) = 70 \text{ Å}$ or $d(11\overline{3}) = 60 \text{ Å}$.

Comparisons between the two other faults is not easy because the mismatch density is sensibly the same ($4 a^2$ for the $(001)$ fault versus $3.6 a^2$ for the $(11\overline{3})$ one), but their distribution between $T-T$ and $T-O$ mismatches is quite different. However it has to be remarked that for non equimolar spinel $n = 1.8$ the density of structural cation vacancies is $1$ vacant site per $a^3$ volume and without considering any possible cation vacancy ordering within the fault planes, the density of $T-O$ mismatches for the $(11\overline{3})$ fault dramatically falls down by more than $83\%$ ($0.2$ versus $1.2 a^2$). The fault energy should decrease correspondingly if the $T-O$ mismatches are the most energetic ones (they correspond to a much larger shortening of the ideal cation-cation distance than $T-T$ mismatches). A much lower fault energy for $(11\overline{3})$ would result.

In conclusion, the non conservative (but neutral) faults in $\{ 110 \}$, $\{ 100 \}$ and $\{ 113 \}$ account for all the observed preferred orientations of the dislocations left by a slight plastic strain at high temperature.

The screw dislocations observed in $\{ 111 \}$ glide are not seen dissociated. If dissociated, their dissociation plane has to be one of the above planes, otherwise this new fault plane would lead to new preferred orientations in other cases (for instance for $\{ 110 \}$ glide). Moreover, recently Veyssière et al. $[17]$ have been able

$^{(2)}$ Note that $(11\overline{2})$, $(11\overline{4})$, $(11\overline{3})$ are very near to $(11\overline{3})$ (see Fig. 4d), and the experimental measurements of $\delta$, the projected dissociation width on the micrograph plane, are not precise enough to allow distinction between these cases to be done. However, a fault in $(11\overline{2})$, $(11\overline{4})$ or $(11\overline{3})$, with a displacement vector $R = a/4 [01\overline{1}]$ (the partial Burgers vector of the actual dislocation) would not be neutral (see Appendix). Therefore, the case reported on figure 4 unambiguously corresponds to a climb dissociation in $(11\overline{3})$ plane, with a true dissociation width of $60 \text{ Å} \pm 10\%$. 


to observe in weak-beam conditions that screws produced by deformation at low temperature under high confining pressure are very slightly dissociated in \{100\} planes.

Therefore the three fault types mentioned above \{100\}, \{110\} and \{113\} are necessary and sufficient to account for all the observations of low mobility or even sessile orientations known until now for plastic glide in spinel. At very high temperature or for very large \(n\), when climb becomes the prominent deformation mechanism, new climb dissociations seem to occur (in \{112\} for instance [7]) but clearly such dissociations do not operate in glide.

**Appendix.** — A fault is characterized by its fault plane \((hkl)\) and its displacement vector \(\mathbf{R}\). One only considers here the non conservative faults i.e. \(\mathbf{R}\) is off

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**Fig. 5.** — Schematic representation of the three non conservative faults: — large circles are octahedral cations (Al in normal spinel), small circles are tetrahedral cations (Mg); oxygen anions are represented by black dots; — the height of cations above the projection plane are indicated by the position of a radius in the circle : for a cation in the projection plane : \(0\); for a cation at the height \(u, p/q (u = \text{unit height})\), the radius is rotated by an angle of \(2\pi p/q\) (anticlockwise rotation) \(\Theta\).

- **a)** Edge dislocation dissociated by climb; projection on (001) plane, unit height \(u = a\). The dislocation line is parallel to [001], \(b = a\sqrt{2}[110]\). The dissociation plane is (110). Across the fault plane, only some cations are in faulted configurations: \(T\) and \(T'\) are too near one to the other. These mismatched cations are hatched.

- **b)** 60° dislocation dissociated in (001); projection on (110), unit height \(u = a\sqrt{2}/2\). The dislocation line is parallel to [110], \(b = a/2[011]\). Only one partial dislocation is represented. Across the fault plane, the distances between two tetrahedral cations (\(T - T'\)) and between adjacent tetrahedral and octahedral cations (\(T - O'\)) are faulted. c) Fault \(a/4[011](113)\), projection on (110), unit height \(u = a\sqrt{2}/2\). The shortest (faulted) distances \(T - T'\) and \(T - O'\) are the same as in the case \(a/4[011](001)\) but the density of \(T - O'\) mismatches is lower.
The (hkl) plane. Creating a fault consists in removing (or adding) a crystal layer with a thickness $e = \frac{\mathbf{R}.\mathbf{n}}{|\mathbf{n}|}$ where $\mathbf{n} = q[hkl]$ ($a = \text{parameter of the cubic unit cell}$) is the normal to the fault plane.

The crystal structure can be decomposed in elementary layers parallel to (hkl). Let $e_0$ be the minimum thickness of such layers with the nominal chemical composition (MgAl$_2$O$_4$ for equimolar spinel). Furthermore let $P = e/e_0$ be the number of layers to be removed (or added) for the fault creation. The fault is neutral and keeps the composition unchanged only if $P$ is an integer.

A detailed examination of the spinel structure leads us to the following results (Table A.1).

One notes that for a displacement vector $\mathbf{R} = a/4 < 110 >$ and a fault plane $\{001\}$, $P$ is either 1 or 0 (in this latter case the fault is conservative). Consequently any fault of the type $a/4 < 110 > \{001\}$ is a neutral one and the chemical composition is not changed. The various parameters characterizing other possible faults are reported in table A. II.

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Table A. I

<table>
<thead>
<tr>
<th>Fault plane</th>
<th>${001}$</th>
<th>${110}$</th>
<th>${111}$</th>
<th>${112}$</th>
<th>${113}$</th>
<th>${140}$</th>
<th>${114}$</th>
<th>${115}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$e_0$</td>
<td>$a/4$</td>
<td>$\sqrt{2}/4$</td>
<td>$\sqrt{3}/3$</td>
<td>$a/6/12$</td>
<td>$a/11/11$</td>
<td>$a/17/68$</td>
<td>$a/2/12$</td>
<td>$a/27/27$</td>
</tr>
<tr>
<td>$P$</td>
<td>$\frac{4}{a^2}$</td>
<td>$\frac{2}{a^2}$</td>
<td>$\frac{2}{a^2}$</td>
<td>$\frac{4}{a^2}$</td>
<td>$\frac{2}{a^2}$</td>
<td>$\frac{2}{a^2}$</td>
<td>$\frac{4}{a^2}$</td>
<td>$\frac{2}{a^2}$</td>
</tr>
</tbody>
</table>

Table A. II

<table>
<thead>
<tr>
<th>Fault plane</th>
<th>$\langle a/4, 110 \rangle {001}$</th>
<th>$\langle a/4, 110 \rangle {110}$</th>
<th>$\langle a/4, 110 \rangle {111}$</th>
<th>$\langle a/4, 110 \rangle {112}$</th>
<th>$\langle a/4, 110 \rangle {113}$</th>
<th>$\langle a/4, 110 \rangle {114}$</th>
<th>$\langle a/4, 110 \rangle {115}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$P$</td>
<td>1</td>
<td>1/2</td>
<td>1/2</td>
<td>1/2</td>
<td>1/2</td>
<td>1/2</td>
<td>1/2</td>
</tr>
<tr>
<td>$\langle \mathbf{R}, \mathbf{n} \rangle$ angle</td>
<td>$45^\circ$</td>
<td>$60^\circ$</td>
<td>$35^\circ$</td>
<td>$55^\circ$</td>
<td>$73^\circ$</td>
<td>$30^\circ$</td>
<td>$32^\circ$</td>
</tr>
<tr>
<td>Chemical composition of the removed layer</td>
<td>MgAl$_2$O$_4$</td>
<td>[AlO$_2$]$^-$ or [AlMgO$_2$]$^+$</td>
<td>[AlO$_2$]$^-$ or [AlMgO$_2$]$^+$</td>
<td>[AlO$_2$]$^-$ or [AlMgO$_2$]$^+$</td>
<td>[AlO$_2$]$^-$ or [AlMgO$_2$]$^+$</td>
<td>[AlO$_2$]$^-$ or [AlMgO$_2$]$^+$</td>
<td>[AlO$_2$]$^-$ or [AlMgO$_2$]$^+$</td>
</tr>
<tr>
<td>Experimental characterization</td>
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<td>observed</td>
<td>observed</td>
<td>observed</td>
<td>observed</td>
<td>observed</td>
</tr>
</tbody>
</table>

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The values $\langle \mathbf{R}, \mathbf{n} \rangle$ are given by the entries $\mathbf{R}$ and $\mathbf{n}$ in Table A. I. The chemical composition of the removed layer is determined in the following manner:

1. For $\langle a/4, 110 \rangle \{001\}$, the layers are isotropic.
2. For $\langle a/4, 110 \rangle \{110\}$, the layers are anisotropic.
3. For $\langle a/4, 110 \rangle \{111\}$, the layers are anisotropic.
4. For $\langle a/4, 110 \rangle \{112\}$, the layers are anisotropic.
5. For $\langle a/4, 110 \rangle \{113\}$, the layers are anisotropic.
6. For $\langle a/4, 110 \rangle \{114\}$, the layers are anisotropic.
7. For $\langle a/4, 110 \rangle \{115\}$, the layers are anisotropic.

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References