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Materials Science and Engineering A 400–401 (2005) 283–293

MATERIALS SCIENCE & ENGINEERING A

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Transmission electron microscopy study of dislocation motion in icosahedral Al–Pd–Mn

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Received 13 September 2004; received in revised form 6 December 2004; accepted 28 March 2005

Abstract

Perfect and imperfect dislocations trailing phason faults in quasi-crystals are introduced using a simplified two-dimensional aperiodic structure. Then, on the basis of observations of deformed specimens as well as in situ experiments in a transmission electron microscope, the motion of dislocations in icosahedral Al–Pd–Mn is shown to take place exclusively by climb. Under such conditions, the very high brittleness of Al–Pd–Mn at low and medium temperatures is proposed to be a consequence of the difficulty of glide, which itself appears to be an intrinsic property of the quasi-crystalline structure.

Keywords: Quasi-crystals; Phason faults; In situ transmition detection microscopy

1. Introduction

The mechanical properties of quasi-crystals are different from those of ordinary crystalline alloys in several aspects. In particular, they remain highly brittle up to 3/4 of their melting temperature but become very ductile above. Overviews of the mechanical properties of quasi-crystals with various structures have been made by Takeuchi [1] and Feuerbacher et al. [2]. The most extensively studied quasi-crystals are Al–Cu–Fe and Al–Pd–Mn with the icosahedral structure [see e.g. Brunner et al. [3], Geyer et al. [4], Giacometti et al. [5], Kabutoya et al. [6]). In constant strain-rate deformation, they are characterised by a pronounced yield drop followed by a low (often negative) hardening stage, small activation volumes, and high activation energies. Since the work of Wollgarten et al. [7], it is known that deformation proceeds by dislocation motion, at least in Al–Pd–Mn. However, the exact origin of the difference between quasi-crystals and crystals is still an open question. This paper is aimed at (i) introducing dislocations in quasi-crystals and (ii) presenting recent results leading to new explanations for the sharp brittle-to-ductile transition of Al–Pd–Mn.

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doi:10.1016/j.msea.2005.03.051

2. Schematic description of dislocations is quasi-crystals

2.1. Quasi-periodic structures

Quasi-periodic structures can be obtained by the cut and projection of a periodic structure with a higher number of dimensions than the final one. The simplest quasi-periodic structure can be derived from a square grid cut along a direction with an irrational slope (Fig. 1(a)). The sides of the truncated squares, projected on the cut line, define an aperiodic one-dimensional sequence of large (L) and small (S) segments. When the slope of the cut line is $1/\tau$, where τ is the golden mean $\left(\tau = 2\cos\frac{\pi}{5} = \frac{1+\sqrt{5}}{2}\right)$, it is called the Fibonacci sequence. The cut line is the physical space containing the aperiodic sequence, and the line perpendicular to this direction and going through the origin (along which the projection is made) is called "perpendicular space". Each node of the square grid has one component in the physical space and one component in the perpendicular space. Fig. 1(b and c) shows the result of a local distortion of the square grid. Along the direction of the physical space, it induces small changes in the positions and lengths of both types of segments, which is equivalent to a local elastic strain field. Along the direction of the perpendicular space, it induces local reversals in

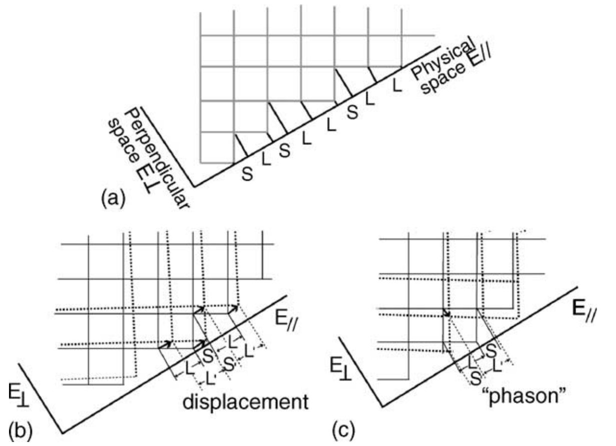

Fig. 1. Properties of a one-dimensional aperiodic sequence. (a) Definition by cut and projection of a two-dimensional periodic network. (b and c) Strain and phason fields induced by displacements of the periodic network along the arrowed directions.

the sequence of long and short segments. Such local sites of disorder are called "phasons".

A two-dimensional aperiodic tiling can be defined in the same way by the cut of a three-dimensional periodic lattice, as shown in Fig. 2(a). Indeed, the cut can be seen either as a truncated pile-up of cubes, or as an aperiodic planar tiling of three different tiles. Note that there is an apparent local three-fold symmetry because the cut plane is close to $\{1\,1\,1\}$. A real three-fold symmetry would be obtained using a $\{1\,1\,1\}$ cut plane, but the resulting two-dimensional tiling would of course be periodic. The tiling contains three families of wavy bands of constant thickness, parallel in average, corresponding to the cut of the three families of dense planes parallel to the three sides of the cubes. One band is underlined in Fig. 2(b). These bands are comparable to the corrugated dense planes of the icosahedral structure.

In the same way, the icosahedral structure of Al–Pd–Mn and Al–Cu–Fe can be defined as the cut and projection of a six-dimensional hypercubic lattice. Fig. 3 shows a cut of this six-dimensional lattice, containing one line of the physical space, along a five-fold direction (noted $A_{5//}$), and one line of the perpendicular space (noted $A_{5\perp}$). The nodes are deco-

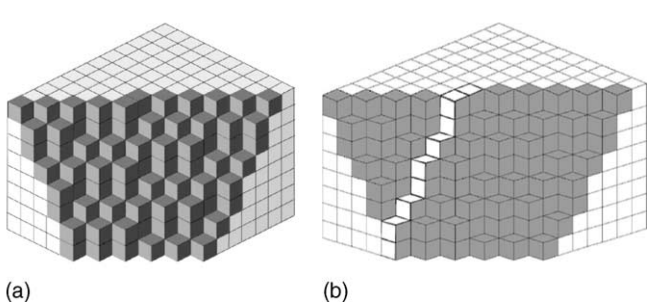

Fig. 2. Two-dimensional aperiodic tiling defined by cut and projection of a three-dimensional periodic cubic lattice. (a) Truncated cubic lattice. (b) Same structure after projection in the cut plane. Note the wavy band corresponding to the emergence of a dense plane.

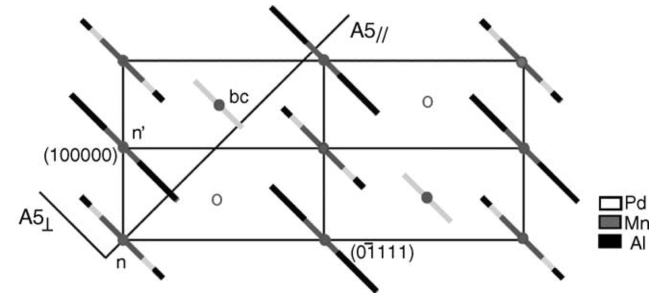

Fig. 3. Section of the six-dimensional hypercubic lattice from which the icosahedral structure of Al–Pd–Mn is deduced. $A_{5//}$ is a five-fold direction of the physical space (courtesy of D. Gratias).

rated with atoms elongated in the direction of the perpendicular space. Atoms intersecting the line of the physical space are really present in the icosahedral structure. They can however be replaced by neighbouring ones if the six-dimensional lattice is locally displaced along the perpendicular direction, thus introducing a phason similar to that shown in Fig. 1(c).

It can be noted that the number of unit vectors necessary to describe all the nodes with integer coordinates is equal to the number of dimensions of the corresponding periodic lattice (two for the Fibonacci sequence of Fig. 1, three for the two-dimensional tiling of Fig. 2, and six for the icosahedral structure).

2.2. Dislocations: a simplified description

The main properties of dislocations in quasi-crystals can be understood using the two-dimensional tiling of Fig. 2. A dislocation can be introduced by the Volterra process as in crystals. However, this can be made either after the cut, in the aperiodic structure in the physical space, or before the cut, in the periodic structure of higher dimensions.

Fig. 4a shows how a dislocation can be introduced by the first process. A wavy dense row, such as that underlined in Fig. 2b, has been partly removed, and the two lips have been joined together. It seems at a first sight that no stacking fault is introduced during this process, because the two half structures fit together.[1] This is however not true, as shown in Fig. 4b. Indeed, when the relief is restored, it becomes obvious that the tiling around the dislocation cannot anymore be defined by the cut of the three-dimensional structure by a single plane. A step appears along the cut, which indicates that the Volterra process has introduced a stacking fault characterised by a displacement in the direction perpendicular to the figure (i.e. the direction of the perpendicular space). The dislocation, which has a Burgers vector $b_{//}$ contained in the physical space, is thus imperfect. Since $b_{//}$ is the projection of a three-dimensional translation vector B corresponding to one edge of the cubes, the displacement across the fault is

[1] Note however that a shear displacement would have been much more difficult to accomodate (see Section 5).

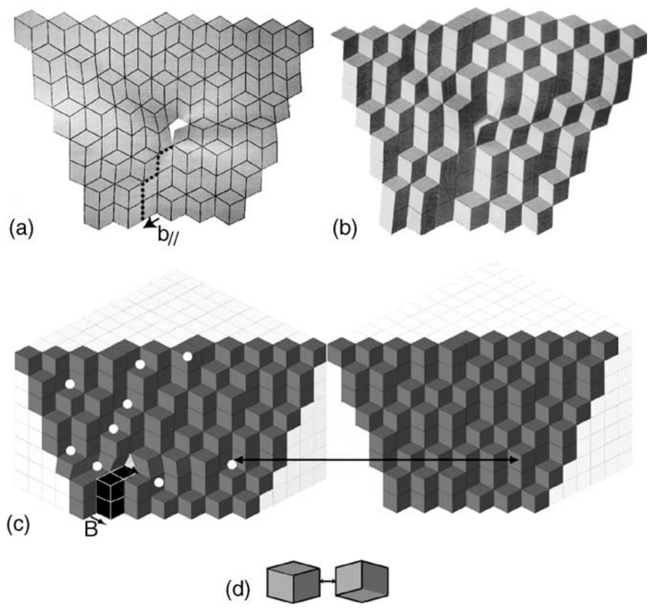

Fig. 4. Dislocations in the two-dimensional tiling of Fig. 2. (a and b) The Volterra process is performed after the cut and projection, in the plane of the tiling (physical space). (c) The Volterra process is performed before the cut and projection, in the three-dimensional space. White dots correspond to phasons introduced by the dislocation. (d) Individual phason.

either $b_{//}$ in the physical space, or $b_\perp = B - b_{//}$ in the perpendicular space. This shows that the displacement vector of a stacking fault is defined modulo a translation vector of the periodic lattice, as in crystals.

Fig. 4(c) shows a dislocation introduced by a Volterra process in the periodic three-dimensional structure. An extra plane has been introduced in *B*. Then, the dislocated three-dimensional lattice has been truncated by the plane of the physical space, and projected on this plane. The resulting two-dimensional structure contains a dislocation, which appears perfect as no stacking fault can be seen. However, since the nodes of the three-dimensional lattice have been slightly displaced in the direction perpendicular to the figure (namely the direction of the perpendicular space) by the insertion of *B*, the plane of the physical space does not intersect the same cubes, i.e. some cubes appear to be either added or removed with respect to the original structure (compare the two schemes Fig. 4(c)). This is equivalent to a field of individual phasons such as that described in Fig. 4(d) (and equivalent to that introduced in Fig. 1(c)), all around the dislocation. The dislocation Burgers vector is *B*, with an elastic component $b_{//}$ (equal to that of the imperfect dislocation in Fig. 4(a)), and a phason component $b_\perp$. In other words, the step introduced in Fig. 4(b) has been smoothed and transformed into a helicoidal slope centred at the dislocation, which means that the phasons condensed in the stacking fault have been dispersed.

Imperfect dislocations are formed at low temperatures. They can transform into perfect ones at high temperature when the phasons are sufficiently mobile to diffuse in the icosahedral lattice. These notions can be extended to systems where the periodic lattice has more than three-dimensions.

2.3. Properties of dislocations in Al–Pd–Mn

Dislocations have been observed and analysed for the first time by transmission electron microscopy in Al–Cu–Fe by Devaud–Rzepsky et al. [8] and Elabard and Spaepen [9]. Subsequent determinations of Burgers vector and dislocation contrasts have been made by Zhang et al. [10], Wollgarten et al. [11], Dai [12], and Wang and Dai [13]. Dislocations in Al–Pd–Mn have been studied later by Dai [14], Feng et al. [15], and Wollgarten et al. [7]. The number of line splitting in convergent beam electron diffraction, and the visibility criteria in bright field images, are directly related to the phase shift introduced by the dislocations. This phase shift is $g_{//}\cdot b_{//}$ for imperfect dislocations, and $G.B = g_{//}\cdot b_{//} + g_\perp\cdot b_\perp$ for perfect ones. The second one is an integer, and the former one is irrational but generally close to an integer value. The rules of contrast of perfect dislocations have been established by Wollgarten et al. [16]: they are visible for $G.B \neq 0$ but out of contrast for $G.B = 0$, which can happen in two cases, (i) $g_{//}\cdot b_{//} = 0$ and $g_\perp\cdot b_\perp = 0$ (strong extinction condition) and (ii) $g_{//}\cdot b_{//} = -g_\perp\cdot b_\perp \neq 0$ (weak extinction condition). In the latter case, the phase shift introduced by the strain field ($g_{//}\cdot b_{//}$) is exactly compensated by the phase shift introduced by the phason field ($g_\perp\cdot b_\perp$), which occurs for a single diffraction spot of a diffraction pattern row (see Fig. 5(e and f)). In the former case, the dislocation is invisible for all the diffraction spots of the same row (Fig. 5(c and d)). As shown by Caillard et al. [17], phason faults obey the same rules of contrast as stacking faults in crystals, based on the value of either $g_{//}\cdot b_{//}$ or $g_\perp\cdot b_\perp$. A summary of all rules of contrast can be found in Mompiou et al. [18].

The role of dislocations in the plasticity of Al–Pd–Mn has been demonstrated by Wollgarten et al. [7]. Subsequent experiments have shown that the density of dislocations reaches a maximum at around 5% of deformation after the yield point, and surprisingly decreases above (Schall et al. [19]). In a first time, the mode of dislocation motion has not been studied experimentally because Burgers vectors and planes of motion could not be determined simultaneously. Dislocations have been then considered to move by glide, i.e., in a plane containing $b_{//}$. Takeuchi and Hashimoto [20] have proposed a Peierls-type controlling mechanism that they still considered valid recently (Takeuchi [21]). Glide was also assumed to be the only mode of motion by Rosenfeld et al. [22]. A glide process controlled by the crossing of localised obstacles (Mackay clusters) has been subsequently proposed by Feuerbacher et al. [23], and improved by Messerschmidt et al. [24]. Climb has been considered as a possible rate-controlling mechanism at very high temperatures ($T > 800\,^\circ$C in Al–Pd–Mn) in a publication by Brunner et al. [25] but not in a further one (Brunner et al. [3]). Some climb has also been obtained in atomistic simulations of Dilger et al. [26].

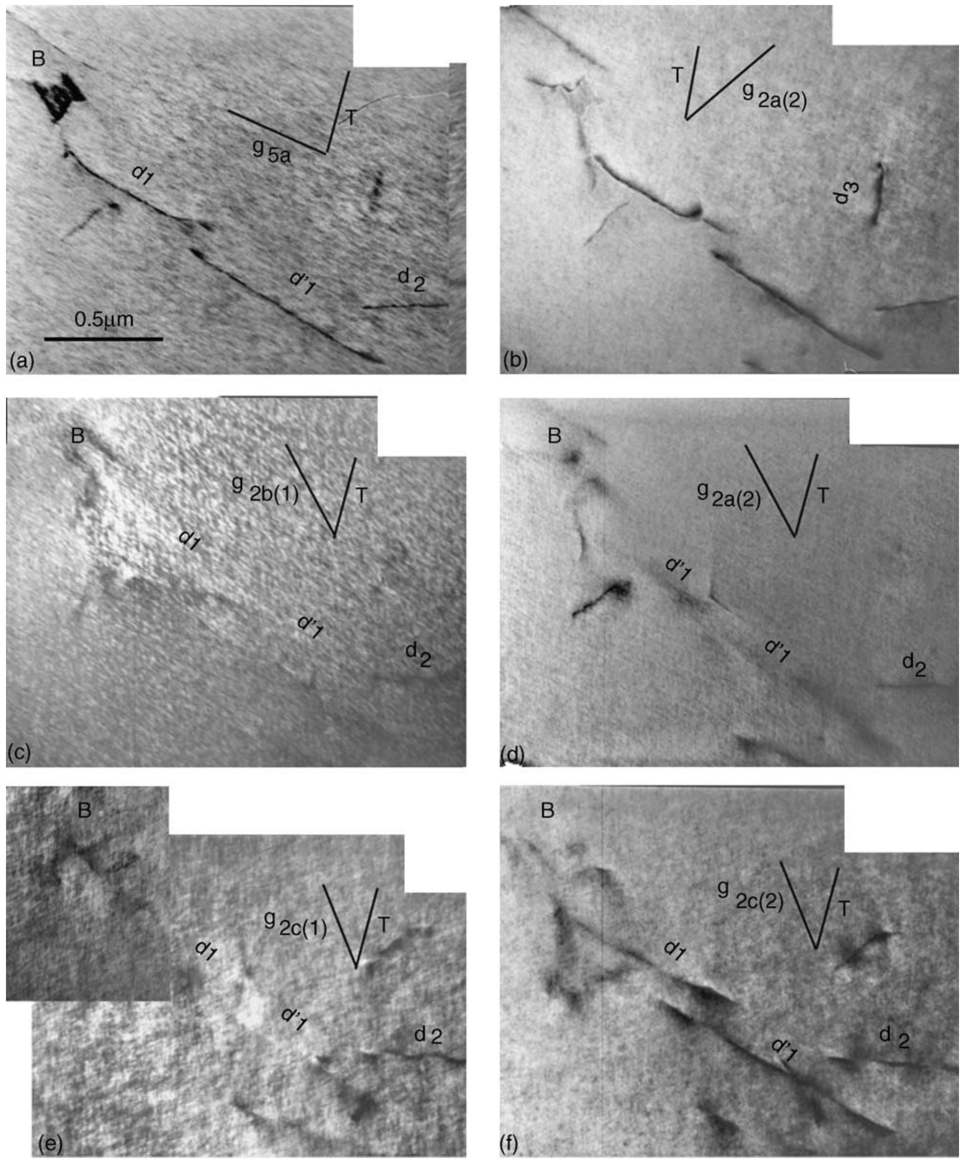

Fig. 5. Perfect dislocation seen under different diffracting conditions (from Caillard et al. [36]). (a) Single contrast, (b) double contrast on d_1 and d'_1. (c–d) Strong extinction on d_1 and d'_1 for two parallel diffraction vectors. (e) Weak extinction on d_1 and d'_1. (f) Single contrast on d_1 and d'_1 for a diffraction vector parallel to the preceding one, but τ times larger.

In spite of these emerging ideas, analyses of phason faults with displacement vectors perpendicular to their plane have not been attributed to a dislocation climb motion but to some rearrangement of the structure after glide (Wang et al. [27]). Climb has been subsequently evoked as a possible recovery process controlling the high temperature range of plasticity by Schall et al. [19] and by Messerschmidt et al. [28]. In the latter publication, it was also considered that climb may control the dislocation density, but that dislocation motion may essentially take place by glide. A model of plasticity by Feuerbacher et al. [29] explains successfully the mechanical properties on the basis of dislocation motion by glide and recovery by cross-slip.

The first simultaneous determinations of Burgers vectors and planes of motion have been made by Caillard et al. [17,30]. They revealed an extensive dislocation motion by pure climb in an as-grown Al–Pd–Mn ingot presumably deformed under the thermal stresses generated by the cooling process. Plastic deformation by pure climb has been confirmed by Mompiou et al. [18], Caillard et al. [31], Texier et al. [32], and Mompiou and Caillard [33], in Al–Pd–Mn samples deformed between 20 °C and 300 °C, under a high confining pressure.

Climb is now accepted as the principal mode of dislocation motion in Al–Pd–Mn by Messerschmidt and Bartsch [34], but the effective controlling mechanism as well as the exact role of glide are however still debated (Messerschmidt et al. [35]).

3. Observations in Al–Pd–Mn deformed at 300 °C

Al–Pd–Mn single grains have been compressed at 300 °C, well below the brittle-to-ductile temperature, under a high confining pressure (Mompiou et al. [18]). Fig. 6 shows the dislocations present in a foil cut perpendicular to the five-fold direction of the compression axis. Long and wavy dislocations contained in the foil plane are seen in Fig. 6(a). Dislocation d_1 (and several equivalent ones) are out of contrast in Fig. 6(b), and in strong residual contrast in Fig. 6(c and d). Since the diffraction vectors in Fig. 6(b–d) are in the sample plane, it can be inferred that the corresponding Burgers vector is perpendicular to the foil plane, i.e. parallel to the compression axis. Dislocation d_2, out of contrast in Fig. 6(c and d), has another Burgers vector, parallel to a two-fold direction at 58° from the compression axis. Since these dislocations have moved in the foil plane, it is clear that their motion has involved a large component of climb (pure climb for d_1). Fig. 7 shows the same dislocations in a foil cut at 20° from the preceding one. Note the fringe contrast trailed by the pair of dislocations d_1 in the plane perpendicular to the compression axis, which changes across dislocation d_2, and when the sign of the diffraction vector is changed (Fig. 7(a and b)). This fringe contrast is typical of the phason fault trailed by the dislocations at low temperature. It was not seen in Fig. 6 because the faults were parallel to the foil plane. The fringe contrast disappears, and the corresponding dislocations d_1 have a strong residual contrast, for diffraction vectors parallel to the fault plane (Fig. 7(c and d)). This confirms that the Burgers vectors are perpendicular to the fault plane, namely that dislocations d_1 have moved by pure climb. Note that dislocation d_2 (in double contrast in Fig. 7(c), and out of contrast in Fig. 7(d)) has another Burgers vector, but that its motion has also involved a component of climb.

Fig. 6(e) shows dislocations d_1 in residual contrast (thick contrasts in the top-right) and edge-on phason faults trailed by another dislocation family in a two-fold plane parallel to the compression axis (bright lines along trP_3). Determinations of the signs of the corresponding displacement vectors have shown that dislocations climbing in the plane perpendicular to the compression axis have absorbed vacancies, whereas those climbing in planes parallel to the compression axis have emitted vacancies (Mompiou et al. [18]). This shows that an exchange of vacancies has occurred between the two systems. Dislocations absorbing vacancies have been activated by the compression stress, with a high Schmid factor. On the contrary, those moving in planes parallel to (or close to) the compression axis have a zero (or a small) Schmid factor. Their motion was thus probably induced by a chemical stress, due to an under-saturation of vacancies resulting from the motion of the first system.

4. In situ experiments in Al–Pd–Mn

The first in situ experiments have been made by Wollgarten et al. [37]. They revealed straight dislocations parallel to two-fold directions, moving viscously in dense planes perpendicular to two-, three- and five-fold directions. Since the Burgers vectors could not be determined, the dislocations were considered to move by glide.

We have carried out new in situ experiments of two kinds. In a first set of experiments, microsamples were only heated, and the few dislocation movements caused by the rapidly vanishing thermal stress were observed and recorded. The contrast of mobile dislocations has been studied under different diffracting conditions, either during their motion or after quenching in the microscope. In a second set of experiments, microsamples have been strained at high temperature, using a device described by Pettinari et al. [38].

Fig. 8 shows a dislocation frozen by rapid cooling during its motion at 720 °C. It trails two traces on the two surfaces, noted t_1 and t_2. Their direction and variation of separation distance as a function of the tilt angle show that the dislocation has moved in a three-fold plane. The dislocation also trails a fringe contrast, which vanishes progressively, and disappears completely after the dislocation has moved over the distance λ. This contrast is due to a phason fault, as in the preceding section. However, the temperature is high enough to allow the rapid dispersion of the corresponding phasons. The fringes are out of contrast, and the dislocation is in residual contrast, for diffraction vectors parallel to the fault plane (Fig. 8(b and c)). It can thus be inferred that the Burgers vector is perpendicular to the plane of motion, i.e. that the dislocation has moved by pure climb. Other examples of the same process have been published elsewhere (Mompiou et al. [39]).

For slower dislocation movements and/or at higher temperatures, no fringe can be seen in the wake of moving dislocations. This is interpreted as a fast enough dispersion to allow the motion of perfect dislocations. An example of such a behavior is shown in Fig. 9. A straight dislocation parallel to the two-fold direction d moves between pictures (a) and (c). Then, its velocity decreases sufficiently to allow its contrast analysis under several diffracting conditions. It has moved in a five-fold plane which has been deduced from the trace direction trP_5 and the variation of the trace distance between (a)–(c) and (g). It exhibits strong extinctions in (d)–(f) for diffraction vectors contained in the five-fold plane of motion. Here again, the Burgers vector is perpendicular to the plane of motion, showing that the dislocation has moved by pure climb.

Fig. 10 shows a dislocation moving during an in situ straining experiment. It moves to the left in the almost edge-on two-fold plane, with trace trP_2, perpendicular to the straining axis (vertical in the figure). Although contrast analyses have not been made in this case, the motion is likely to occur by pure climb, for which the Schmid factor has its maximum value of 1, rather than by glide, for which the Schmid factor would be zero. Note that this argument is valid because

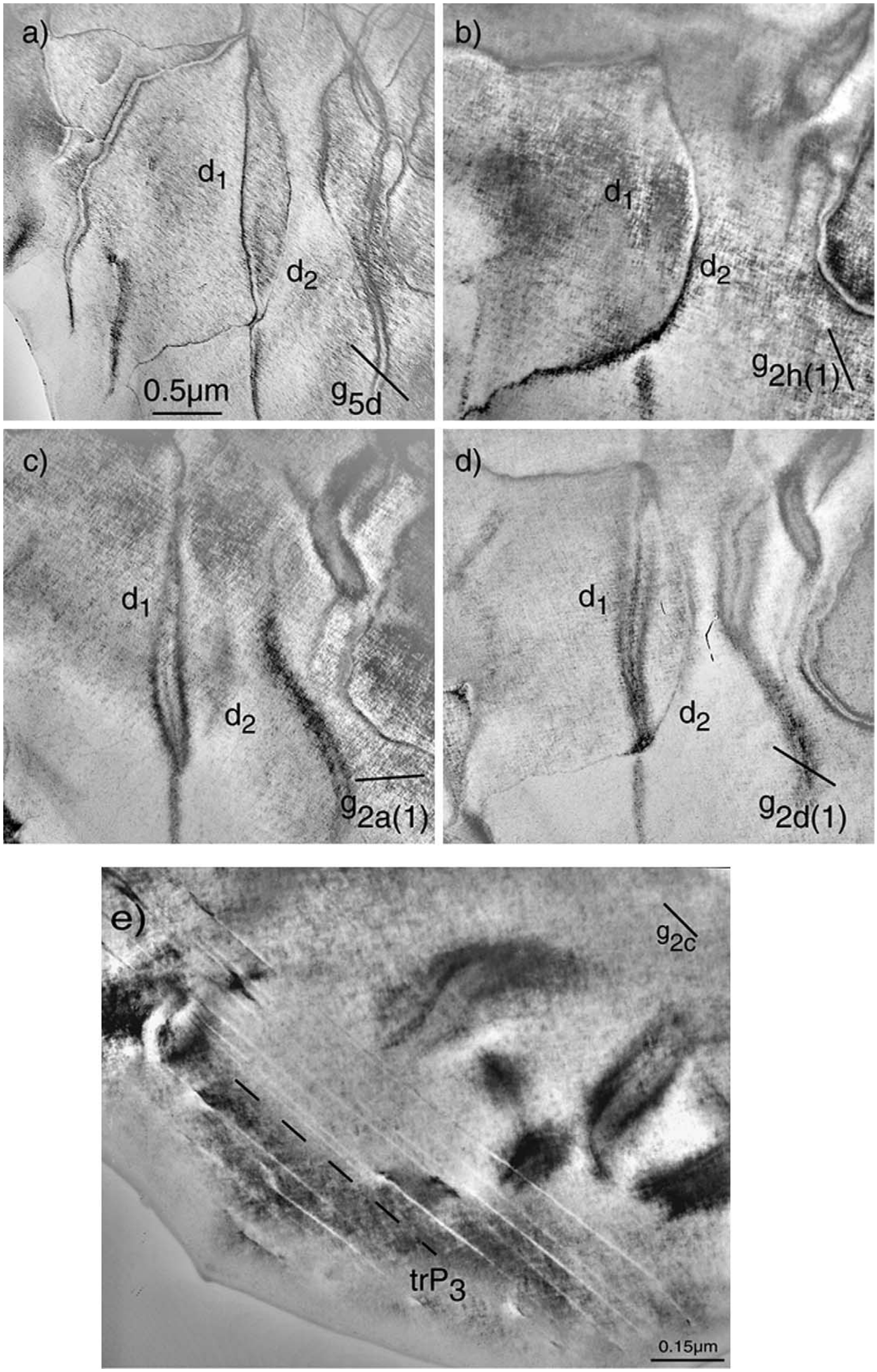

Fig. 6. Dislocations lying in the sample plane, in Al–Pd–Mn deformed at 300 °C under a 7 GPa hydrostatic pressure. Dislocation d_1 is out of contrast in (b), and in residual contrast in (c and d), for two-fold diffraction vectors parallel to the sample plane, and perpendicular to the compression axis. (e) Edge-on phason faults along the direction trP_3, trailed by dislocations d_3 (dislocations d_1 are in residual contrast in the top-right of the picture).

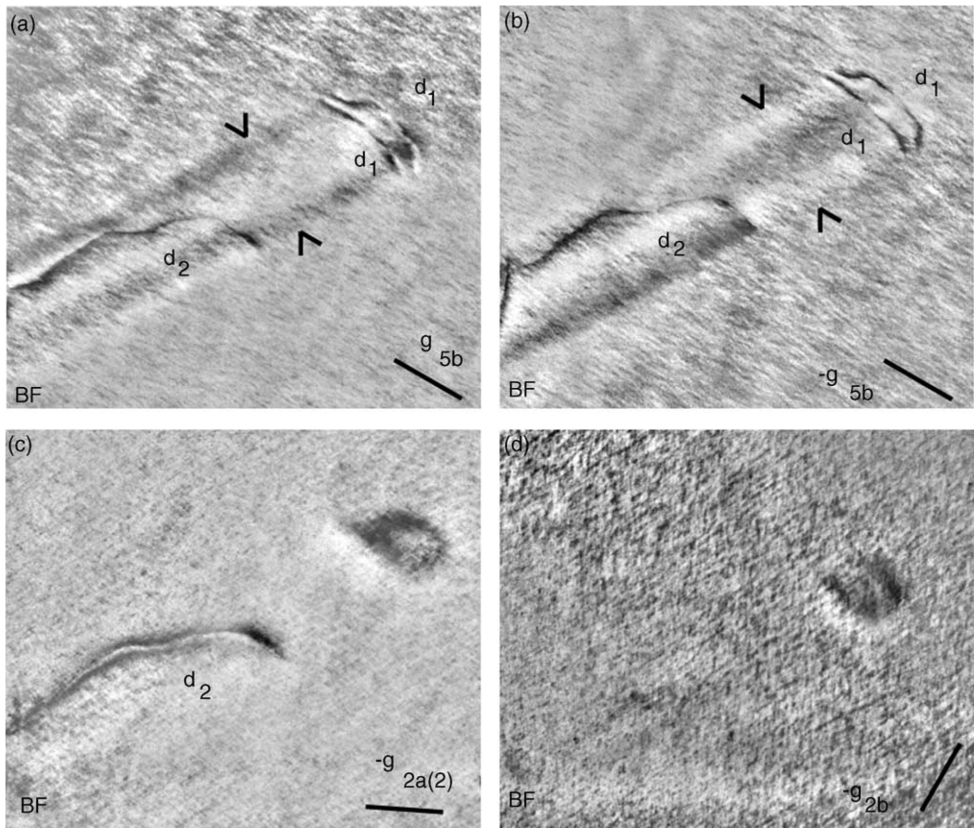

Fig. 7. Same dislocations as in Fig. 6 in a sample plane at an angle of 20° from the preceding one. Note the fringe contrast of the phason fault, which is reversed when the sign of the diffraction vector is changed (pictures a and b), and the residual contrast of dislocations d_1, for two diffraction vectors parallel to the fault plane (pictures c and d).

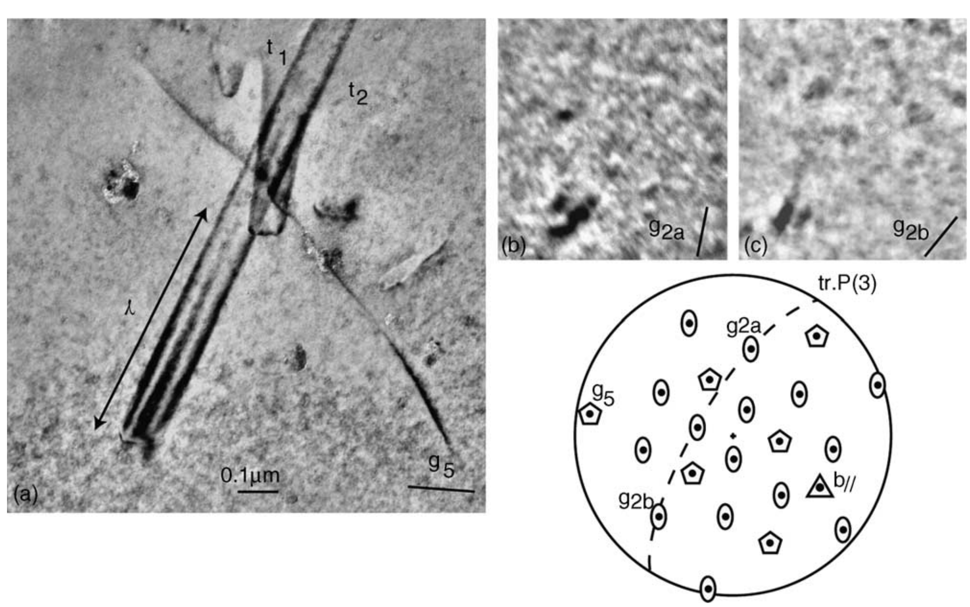

Fig. 8. Phason fault vanishing at a distance λ from an imperfect dislocation frozen during its motion. Extinctions in (b and c) for two diffraction vectors parallel to the fault plane P_3 show that the Burgers vector $b_{//}$ is perpendicular to this plane.

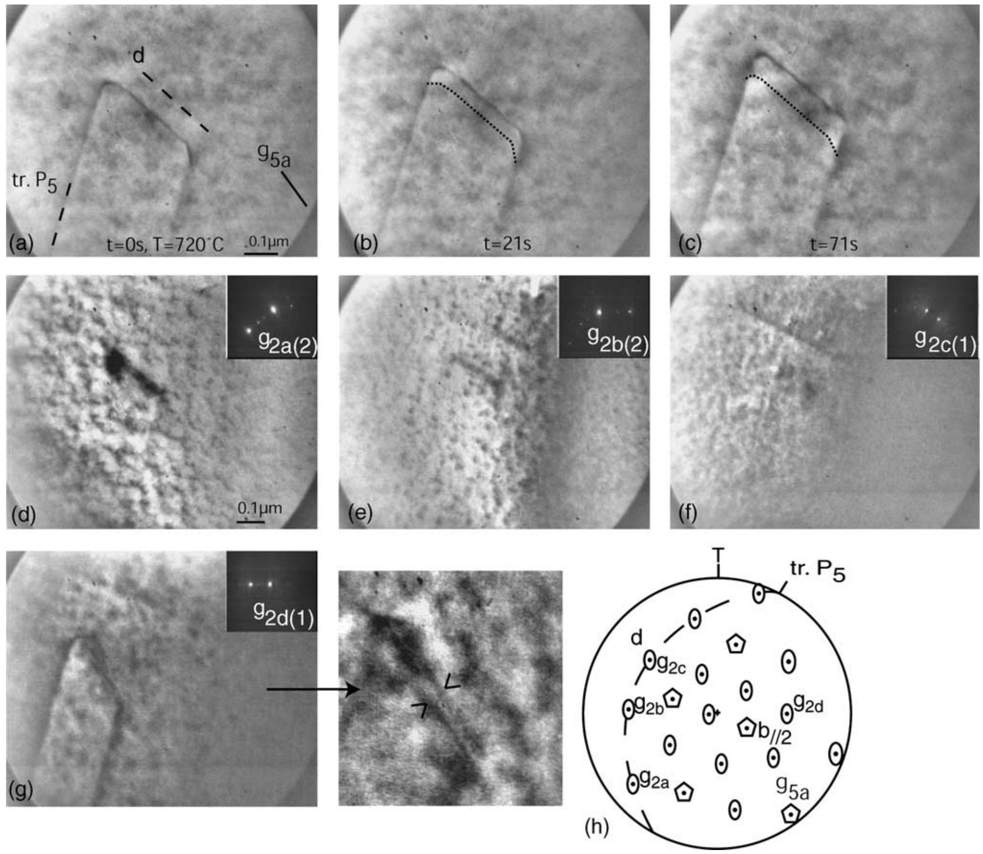

Fig. 9. In situ experiment at 720 °C in Al–Pd–Mn. (a–c) Motion of a perfect dislocation parallel to the two-fold direction d in a five-fold plane. (d–f) Strong extinction conditions showing that the component $b_{//}$ of the Burgers vector is perpendicular to the plane of motion. (g) Double contrast.

the local straining axis in thin foils is closely parallel to the external one in areas where the deformation is concentrated (Couret and Caillard [40], Pettinari et al. [38]).

In the above in situ experiments, all fully analysed dislocations moved by pure climb. Since glide has never been identified, the question of its existence can be addressed. The following experiment yields an answer to this question. Fig. 11(a–e) shows two dislocations with opposite signs approaching each other in parallel five-fold planes (trace trP_5). They interact to form a linear defect, which is identified as a dipole according to its change of contrast when the sign of the diffraction vector is reversed (Fig. 12(a and b)). Since climb is a very fast process at 740 °C, it is very surprising that this dipole does not annihilate, even after a long waiting time of 30 mn, and under an interaction stress of the order of 300 MPa. On the basis of strong extinctions for two diffraction vectors parallel to the five-fold plane of motion, it can be inferred that (as in other situations) the component of the Burgers vector in the physical space is perpendicular to this plane, namely that the two dislocations have moved by pure climb. However, since this Burgers vector is contained in the two-fold plane of the dipole, the reason why annihilation did not take place is that it would have required dislocation motion by pure glide (Fig. 12(e)). Glide is accordingly at least 1000 times a slower process than climb, at least in two-fold planes and at the temperature of the experiment (740 °C).

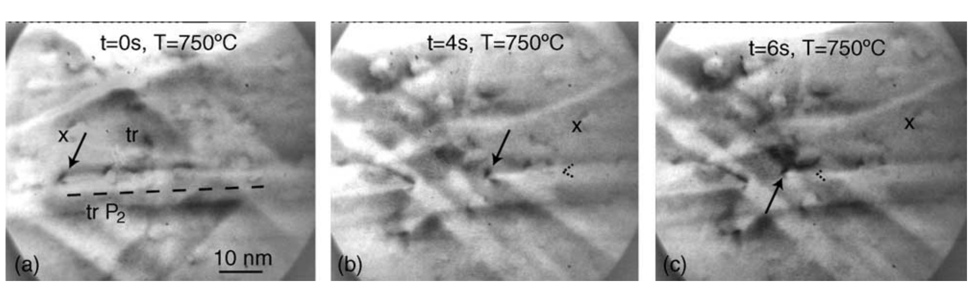

Fig. 10. In situ straining experiment at 750 °C showing the motion of a perfect dislocation (arrowed) in an edge-on plane (trace trP_2) perpendicular to the straining axis. X is a fixed point.

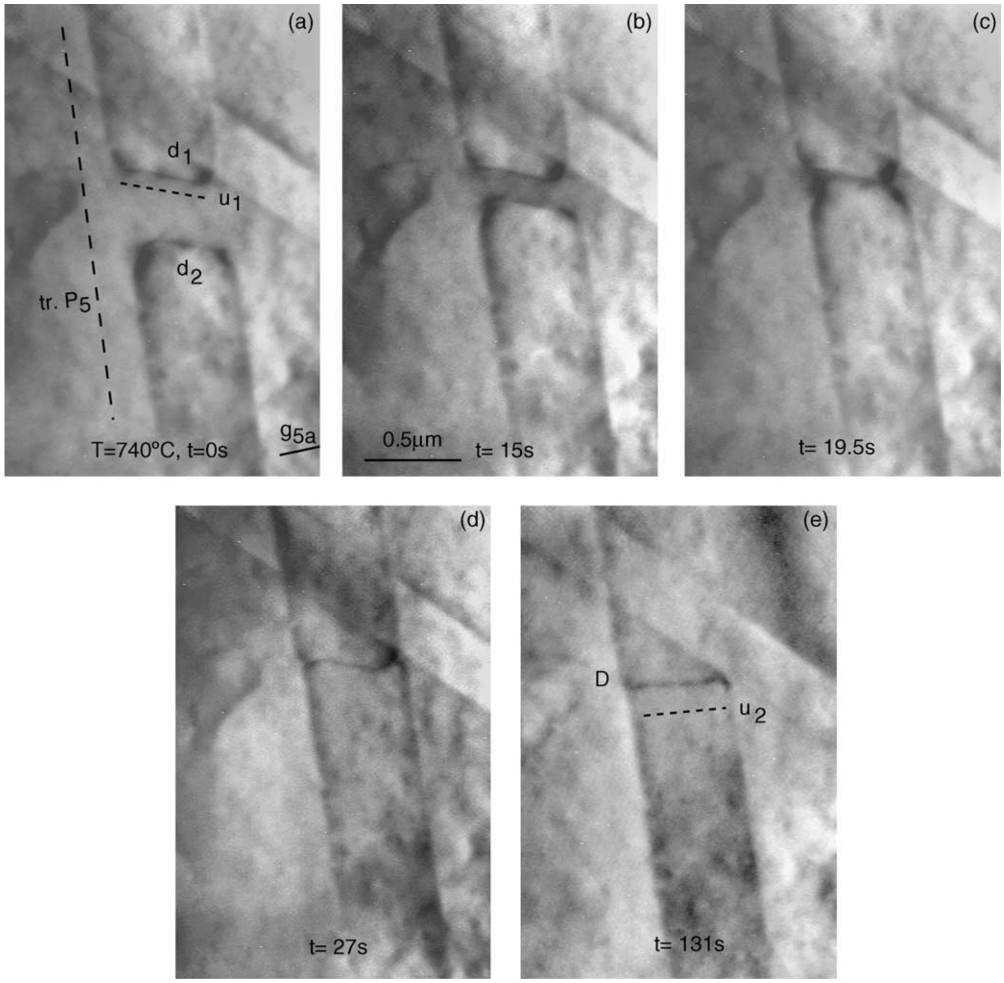

Fig. 11. In situ experiment showing the motion of opposite perfect dislocations (parallel to the two-fold direction u_1), and the formation of a dipole along the pseudo two-fold direction u_2.

5. Discussion

These observations show that climb is the only efficient mode of dislocation motion in Al–Pd–Mn, and that glide is much more difficult, maybe impossible. The difficulty of glide can be related to the high corrugation of the dense planes, which is an intrinsic property of quasi-crystals. As a matter of fact, in the same way as the $\{1\,0\,0\}$ planes of the cubic structure do not end along straight lines in Fig. 2, hyperplanes of the six-dimensional periodic lattice do not end in the physical space along planar structures. Then, shear is likely to induce important disorder in the structure (see e.g. the simulations of Mikulla et al. [41]), and to be accordingly difficult. On the contrary, pure climb by annihilation or duplication of the corrugated dense planes of constant thickness must be an easier process, at least on the basis of topological arguments. As an example, the Volterra process of Fig. 2(b) was easy to perform because it was analogous to a climb mechanism. On the contrary, a shear displacement would have been more difficult to accommodate.

Since climb is also active in crystals, it can be concluded that the main difference between quasi-crystalline Al-Pd-Mn and crystalline metallic alloys is the difficulty of glide in the former material. Since glide is the usual low-temperature plasticity mechanism in crystals, this property is at the origin of the high brittleness of quasi-crystals at low temperatures where climb is too slow a process to accommodate plastic deformation.

In spite of this difference, quasi-crystals exhibit many common properties with semiconductors deforming by glide (e.g. silicon). In both cases, dislocations exhibit straight segments parallel to dense directions, and move viscously. Both materials are brittle at low temperature, and ductile at high temperature where they exhibit a pronounced yield drop. This analogy is not fortuitous. It can be explained because in both cases:

(i) dislocations have a lower energy in deep Peierls valleys;

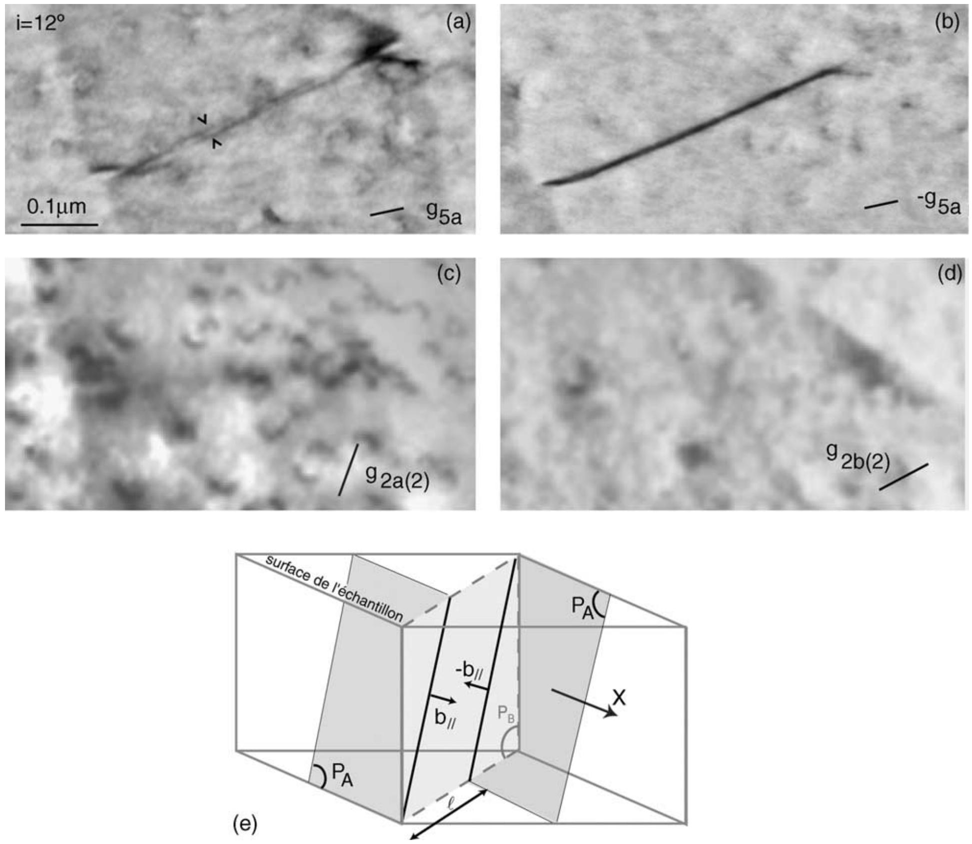

Fig. 12. Contrast analysis of the dipole of Fig. 11e. (a and b) Dipolar effect under $\pm g_{5a}$ conditions. (c and d) Strong extinction conditions showing that the component $b_{//}$ of the Burgers vector is perpendicular to the plane of motion P_A, and parallel to the plane of the dipole P_B. (e) Corresponding scheme.

(ii) they move by a difficult nucleation followed by an easier propagation of kink-pairs (semiconductors) or jog-pairs (Al–Pd–Mn);
(iii) the motion of kinks/jogs along the dislocations is strongly thermally activated, with an activation energy corresponding to the breaking of covalent bonds in semiconductors, and an activation energy of self-diffusion of vacancies in Al–Pd–Mn.

In fact, both types of motion can be described by the theory proposed by Hirth and Lothe [42], which accounts for the small activation volumes (see Caillard and Martin [43] for more details), which in turn contribute to explaining the pronounced yield drops. Dislocation motion in Al–Pd–Mn is however the most complex one on account of (i) the chemical force discussed in Section 3, (ii) the presence of phason faults dissolving more or less rapidly, and (iii) the unknown – and probably complex – definition and diffusion process of “vacancies” in quasi-periodic structures.

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