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Glide and cross-slip of **a**-dislocations in Zr and Ti

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ABSTRACT

Slip systems involving dislocations with <a> Burgers vectors have been studied in hexagonal close packed Zr and Ti, by means of *in situ* straining experiments in a transmission electron microscope, at various temperatures and as a function of resolved shear stress. The results show that Zr and Ti are very similar in many respects. Prismatic slip is activated at rather low resolved shear stresses, and is controlled by the interaction between mobile dislocations and solute atoms (presumably oxygen). Pyramidal slip requires substantially higher resolved shear stresses and is characterized by straight screw dislocations moving by a kink-pair mechanism. Basal slip is activated at and above room temperature, for resolved shear stresses equal or higher than those in the prismatic planes. The slip traces are always wavy, presumably due to intensive cross slip from basal to prismatic planes. It also involves straight screw dislocations moving by a kink-pair mechanism. These microscopic observations are discussed in the light of some aspects of the mechanical behavior, in particular the increase of yield-stress at decreasing temperature and the discontinuity of activation area close to room temperature.

1. Introduction

Secondary slip systems play an important role in the mechanical properties of metals and alloys with the hexagonal close-packed (hcp) structure where primary slip systems are not sufficient to achieve a complete three-dimensional deformation. In the case of zirconium, titanium and their alloys, the easiest (primary) slip system consists of **a**-type 1/3<11−20> dislocations gliding in {1−100} prismatic planes. Secondary slip systems involve the same **a**-dislocations gliding in first order pyramidal {1−101} and basal (0001) planes, as well as **c**+**a** dislocations gliding in pyramidal planes. However, if we restrict to **a**-dislocations, the possible contributions of pyramidal and basal slip to the mechanical properties, remain poorly documented. In particular, the corresponding thermally activated controlling mechanisms are not known.

Previous results and interpretations concerning the different slip systems of **a**-dislocations in Zr and Ti are briefly summarized below. It is known that prismatic slip dominates all the other ones at low temperature. It accounts for the increase of yield stress with decreasing temperature and corresponding small activation volumes. Two different rate-controlling mechanisms have been proposed so far for prismatic slip, i) the pinning of mobile dislocations by oxygen solute atoms [1–7], and ii) a Peierls friction force on screw dislocations [8–13]. The first one is deduced from the strong hardening effect of oxygen, in Ti [5,12] and Zr [3], and the second is supported by several observations of rectilinear screw dislocations, in Ti [12–14], Ti-5.2%Al [15] and Zr [4,16]. We have shown recently that **a**-dislocations can glide in the prismatic planes of Ti and Zr with no friction force along their screw direction, in agreement with ab-initio calculations [17,18]. Similar results have been obtained recently in Ti of various purities [19]. This suggests that the rate-controlling mechanism is the pinning of mobile dislocations by oxygen atoms [15].

Cross-slip onto first-order pyramidal and/or basal planes is expected to become more and more important at increasing temperature. Previous *in situ* observations [13,18] have also shown that **a**-dislocations gliding in prismatic planes can cross-slip in an intersecting pyramidal plane, at 150 K in Ti, but not in Zr. These dislocations most often remain immobile until they cross-slip back into the prismatic plane. However, they can glide steadily and viscously over large distances when the pyramidal plane is sufficiently stressed. Under such conditions, series of double prismatic-pyramidal cross-slip contributes to decrease the average dislocation velocity in the prismatic planes of Ti, and to make prismatic slip jerky ("locking-unlocking" glide process). The prismatic-pyramidal cross-slip, and the high Peierls friction stress in

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pyramidal planes, could also account for the observations of straight screw dislocations in Ti and Zr [12–14].

Cross-slip onto the basal plane has been reported in Zr, at 300 K [16,20], at 78 K but at large deformation [4], and above 850 K [21]. Still in Zr, basal slip must be introduced in modelling for large deformations at room temperature according to [22,23]. In Ti and Ti-5.2%Al, basal slip has been evidenced in pure shear experiments at 200–300 K, and its critical resolved shear stress has been shown to be about three times that of prismatic slip at 300 K [8,15]. Wavy slip involving a component of basal slip has also been observed in *in situ* experiments at room temperature in Ti, when the basal Schmid factor is high [19]. Lastly, basal slip at 300 K is coupled to the observation of straight screw dislocations in Ti-5.2%Al [15].

Other observations have been performed at room temperature, by means of micro-cantilever tests [27], mechanical tests under optical and scanning electron microscope [25], X-ray diffraction [26] and neutron diffraction [24]. They show that basal slip is activated but more difficult than prismatic slip, in Ti, however with a rather large dispersion of the results ($\tau_B/\tau_P \sim 1.1$ in commercially pure Ti with 600 wppm O [24], $\tau_B/\tau_P \sim 1.5$ in Ti with 1600 and 3200 wppm O [25], $\tau_B/\tau_P \sim 1.7$ to 2.1, in commercially pure Ti with 1700 wppm O [26]) and in commercially pure Zr with 2500 ppm Hf and 1000 ppm O($\tau_B/\tau_P \sim 1.2$ [27]). A few ones also conclude that <**a**> pyramidal slip should be 1.2 to 1.4 times more difficult than prismatic slip [24,25].

From this short review, it results that all available data are too fragmentary to be included into crystal plasticity constitutive laws used to compute the overall behavior of polycrystals. In particular, observations have been carried out at a rather large scale, and sometimes only at room temperature, in such a way that they do not give any information concerning the geometry and kinetics of glide of individual dislocations, as well as the corresponding thermally activated controlling mechanisms as a function of temperature. Here we report results of *in situ* experiments in Ti and Zr of various purities, in order to determine i) the conditions for cross-slip from prismatic to pyramidal and basal planes as a function of temperature, ii) the respective roles of Peierls forces and local pinning at solute atoms as a function of slip plane, temperature and solute concentration, and iii) the consequences of these different behaviors on some aspects of the mechanical properties.

Among the specific macroscopic mechanical properties of Zr and Ti discussed in relation with the various involved slip systems, we will emphasize on the steep increase of yield stress with decreasing temperature below room temperature, the huge hardening effect of oxygen, and the discontinuity at room temperature characterized by a peak in the activation area versus temperature curves [3,5,11,12]. This discontinuity is probably related to a change of dislocation behavior, which in Ti has been attributed to an increase of the cross-slip activity above 300 K, presumably in the pyramidal planes [12,14,28].

2. Experimental

A crystal bar of Van Arkel, interstitial free zirconium-hafnium alloy, is available at CEA. It contains only 100 ppm O, 500 ppm Fe, and 2.2% Hf, in weight. It is referred to as the Zr-2Hf alloy in Refs. [29,30]. The alloy has been arc melted under vacuum, followed by 25% rolling at 1033 K, and by 30% cold rolling at room temperature. A final heat treatment was eventually conducted at 973 K during 2 h. Microsamples have been cut by electrical discharge machining, mechanically and electro-chemically polished until electron transparency. They have been strained in a JEOL 2010HC transmission electron microscope using a GATAN low-temperature straining holder and a home-made high-temperature straining holder. Similar results have been obtained in a Zr sponge containing 236 ppm O and 189 ppm Fe, in weight. This material was also arc melted followed by a series of alternate cold rolling and heat treatments.

Other microsamples have been machined out of a recrystallized Zircaloy-4 thin rolled sheet. This alloy contains 1.3% Sn, 0.21% Fe, 0.1% Cr, 0.13% O, in weight. The samples have been strained at room temperature in a TecNai Transmission Electron Microscope using the room temperature Gatan straining holder.

The results in Ti have been obtained long ago in a high-purity single crystal (50 ppm O, 70 ppm Fe) provided by M.P. Biget and G. Saada [31], and in a medium-purity single crystal (3270 ppm O) provided by S. Naka [12,14,28]. They have been partly published in Ref. [13], re-analyzed and compared to those obtained in Zr.

In situ straining experiments in a transmission electron microscope have already been successful in analyzing the behavior of dislocations under stress at various temperatures in metals and alloys. As discussed in the introduction, the knowledge of the stress distribution in the microsample is very important for the interpretation of the results. As shown in Ref. [32], the stress is concentrated and the deformation starts in the two zones where the external tensile direction is tangential to the thin edged hole. Since the corresponding local stress direction is close to the external one, the resolved shear stresses in the different slip systems observed (or not) roughly obey to the Schmid law. This is particularly true at the beginning of plastic deformation, because the local stress direction can change at the vicinity of crack tips forming at large strains or when the plastic zone progressively extends around the hole.

Furthermore, several precautions must be taken to avoid possible thin foil artefacts, in particular when cross-slip must be investigated. Cross-slip can be initiated at free surfaces as a result of line-tension effects, especially when the Burgers vector is close to the foil plane. The same line-tension effects tend to reduce the length of dislocations and rotate them to an edge orientation where no cross-slip is allowed. For these reasons, observations of dislocations with Burgers vectors far from their slip trace directions are preferred.

All crystallographic data necessary to fully interpret the figures are given in Table 1. Slip planes are deduced from the directions and separation distances of the two slip traces left by the gliding dislocations on the two sample surfaces as a function of the tilt angle. A new method for analyzing combined slip systems is used in *in situ* experiments: in case of frequent cross-slip between two planes P_1 and P_2, the slip traces consist of small segments like those described schematically in Fig. 1a, which are often difficult to resolve. However, Fig. 1a shows that if the two traces tr(P_1) and tr(P_2) are oriented in directions consistent with the motion of a given dislocation, the average trace direction corresponding to intensive cross-slip must remain between tr(P_1) and tr(P_2). On the contrary, if a wavy trace has another direction, even locally, cross slip in another plane is necessarily involved. Fig. 1b shows a trace which can be interpreted by an intensive cross-slip between a prismatic (P) and a pyramidal (π_1) plane, and another one which necessarily involves something else, presumably cross-slip in the basal plane as suggested by Fig. 1c.

3. Results

3.1. Zirconium

Experiments below room temperature on nearly pure Zr have already been reported in Ref. [18]. They are briefly recalled below, then new results at and above room temperature are described. Below room temperature planar slip of curved dislocations in prismatic planes is exclusively observed. As shown in Fig. 2, these

Table 1
Crystallographic data.

Figure	Foil plane	Tensile axis	Burgers vector	Prismatic slip planes	Diffraction vector
2	(0–223)	[-936-2]	[-1-120]	(1–100)	(-1103)
3, 4	(2-1-16)	[-6243]	[2-1-10]	(0–110)	(-1101)
6, 7					(1–210)
8	(2-64-3)	[-5140]	[-1-120]	(1–100)	(10-1-1)
9	(-101-5)	[11,-7-4-3]	[-2110]	(0–110)	(11–20)
10	(1–540)	[3-1-22]	[-1-120]	(1–100)	(-1011)
11, 12	(1–540)	[-3121]	[2-1-10]	(0–110)	(-101-1)

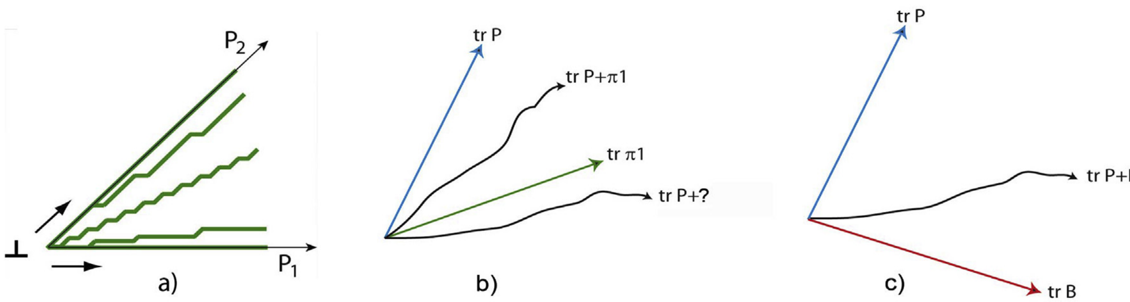

Fig. 1. Scheme of the slip traces left at one sample surface, in case of intensive cross-slip between two elementary slip planes.

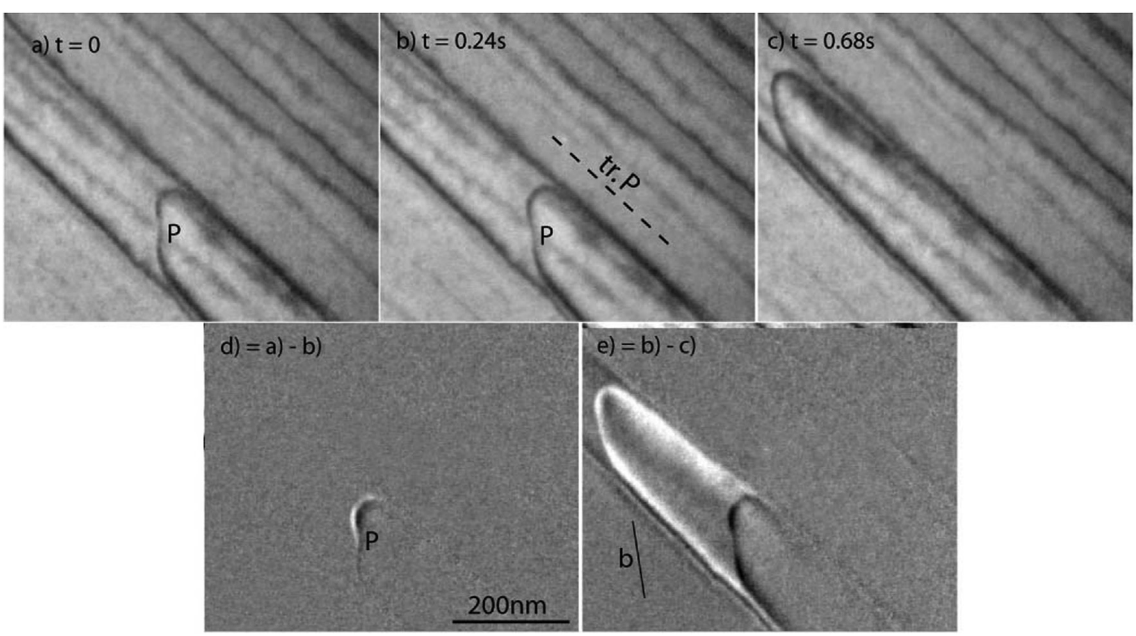

Fig. 2. Prismatic slip in Zr strained at 150 K. The slip trace direction is denoted "trP", b is the Burgers vector direction projected in the observation plane, and P is a pinning point. The two bottom images are subtractions enhancing the dislocation movement. See video as a supplementary material.

dislocations interact with weak pinning points, presumably oxygen atoms. There are neither sessile straight screw segments, nor cross slip into pyramidal planes, contrary to what has been observed in titanium [18].

Supplementary video related to this article can be found at https://doi.org/10.1016/j.actamat.2018.05.038

Basal slip is activated at and above room temperature, sometimes close to hydride precipitates (probably introduced during electro-polishing), and provided the corresponding resolved shear stress is slightly higher than in the prismatic plane (SF(B)/SF(P) = 1.36, see Table 2). Fig. 3 shows three screw dipoles noted 1 to 3 emitted by a hydride noted H. The edge part of dipole 1 has already emerged at the top surface in (a). The corresponding slip trace has a direction close to tr(0001), the trace of the basal plane. The two opposite straight screw dislocations move slowly apart between (a) and (b), as shown by the difference-image (f) = (a) − (b). Then, a second dipole is emitted in (c), and a third one in (e). Other dislocations have glided in the (10-10) prismatic plane in (e). Referring to Fig. 1 and to the trace directions plotted in the bottom-left of Fig. 3, the slip traces along tr.B (or tr.(0001)) cannot result from any combination of prismatic and pyramidal slip. Basal slip is thus clearly activated. The straight aspect and the kinetics of screw dislocations show that they are subjected to a high Peierls friction force when they glide in the basal plane.

Table 2
Elementary slip planes observed as a function of temperature and corresponding Schmid factors (SF) and Schmid factor ratios.

Material	SF(P)	SF(π)/SF(P)	SF(B)/SF(P)	T(K)	Slip planes	Figures
Zr Van Arkel	0.43	1.09 and 0.65	0.53	100–250	P	Fig. 2
	0.25	1.52 and 0	1.36	250	P and onset of B	Not shown
				300–573	P and B	Figs. 3–7
	0.49	0.88 and 0.84	0.06	673	P	Fig. 8
Zr-4 alloy	0.06	3.5 and 1	5.5	300	P and π_1	Fig. 9
Zr-1%Nb-O	0.20	1.95 and 0.25	2.3	523–723	B	Unpublished
	0.49	0.92 and 0.81	0.08	673	P	Fig. 7 of [33]
Ti-50ppmO	0.38	1.21 and 0.63	0.66	150	P	Fig. 2 of [18]
	0.26	1.66 and 0	1.62	150	P and π_1	Fig. 1 of [18]
	0.46	1.04 and 0.72	0.35	150	P (locking-unlocking)	Fig. 4 of [13]
	0.38	1.21 and 0.63	0.66	300	P	Fig. 10
	0.13	2.31 and 0	2.00	300	P and B (and possibly π_1)	Figs. 11–12

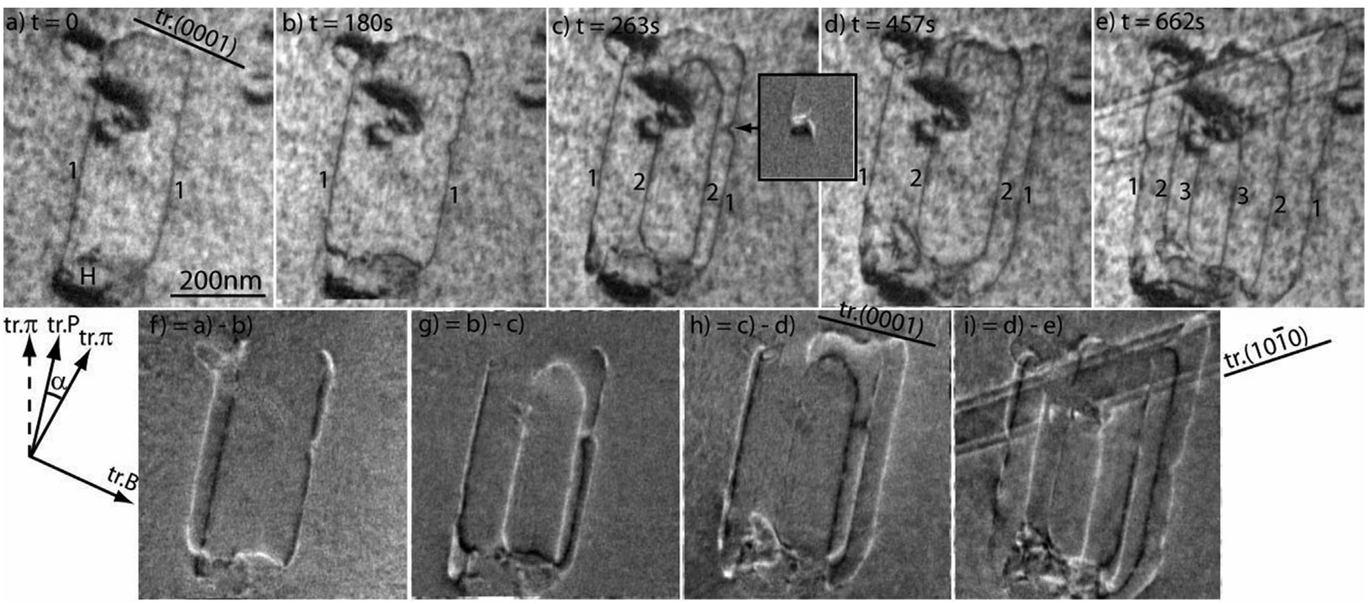

Fig. 3. Basal slip in Zr strained at 300 K. Three screw dipoles (noted 1 to 3) are emitted by a hydride precipitate (noted H). They glide apart and leave a wavy trace of average direction "tr.(0001)". The motion of a super-jog along the screw (or Burgers vector) direction is detailed in the inset. See video as a supplementary material.

Supplementary video related to this article can be found at https://doi.org/10.1016/j.actamat.2018.05.038

In the same microsample, and still at room temperature, Fig. 4 shows the prismatic-basal cross-slip mechanism. Two dislocations move between (a) and (b), as shown by the difference image (c) = (a) − (b). One curved dislocation noted d glides in its prismatic plane with slip trace tr(P), whereas another one noted s glides in the same plane and cross slips in the basal plane with trace tr(B). This second dislocation is straight and screw, and moves slowly and steadily in the basal plane as in Fig. 3. Here again, the slip trace along tr(B) is not rectilinear, which denotes a somewhat wavy glide close to, but not exactly in, the basal plane.

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Slip traces left by a straight screw dislocation in a neighboring area, shown in Fig. 5, are locally almost parallel to the trace of a highly stressed pyramidal plane (SF(π)/SF(P) = 1.52, see Table 1). However, they are not fully rectilinear over long distances as a result of at least some amount of cross-slip in the basal plane (such a cross-slip is clearly visible near the fixed point B). They could be interpreted by as a pyramidal slip mixed with prismatic and basal slip. It is also possible that a mixture of prismatic and basal slip induces similar slip traces. This illustrates the difficulty of isolating unambiguously elementary pyramidal slip planes that is, pyramidal slip which is not the result of an intensive cross-slip between two other slip systems.

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Still in the same microsample but at 373 K, Fig. 6 shows two straight screw dislocations s_1 and s_2 moving steadily between (a) and (b). They trail traces tr(s_1) and tr(s_2) underlined by dots in the difference-image (c). These traces, are slightly wavy but in average parallel to the trace of the basal plane. A component of slip in the basal plane is thus present. The screw motion is faster than at room temperature, in agreement with a strongly thermally activated basal slip.

Supplementary video related to this article can be found at https://doi.org/10.1016/j.actamat.2018.05.038

Increasing again the temperature up to 573 K (Fig. 7), basal slip is still wavy, as shown by the slip traces on both surfaces underlined by dots. The images are highly blurred by the oxidation of the surfaces and by the overlapping of many slip traces, but the dislocation s in (a) and the slip traces in (b) are clearly visible in the difference-image (c). The dislocation in average screw orientation s is more curved and more rapid than at lower temperatures, which indicates that basal slip is almost athermal at 573 K.

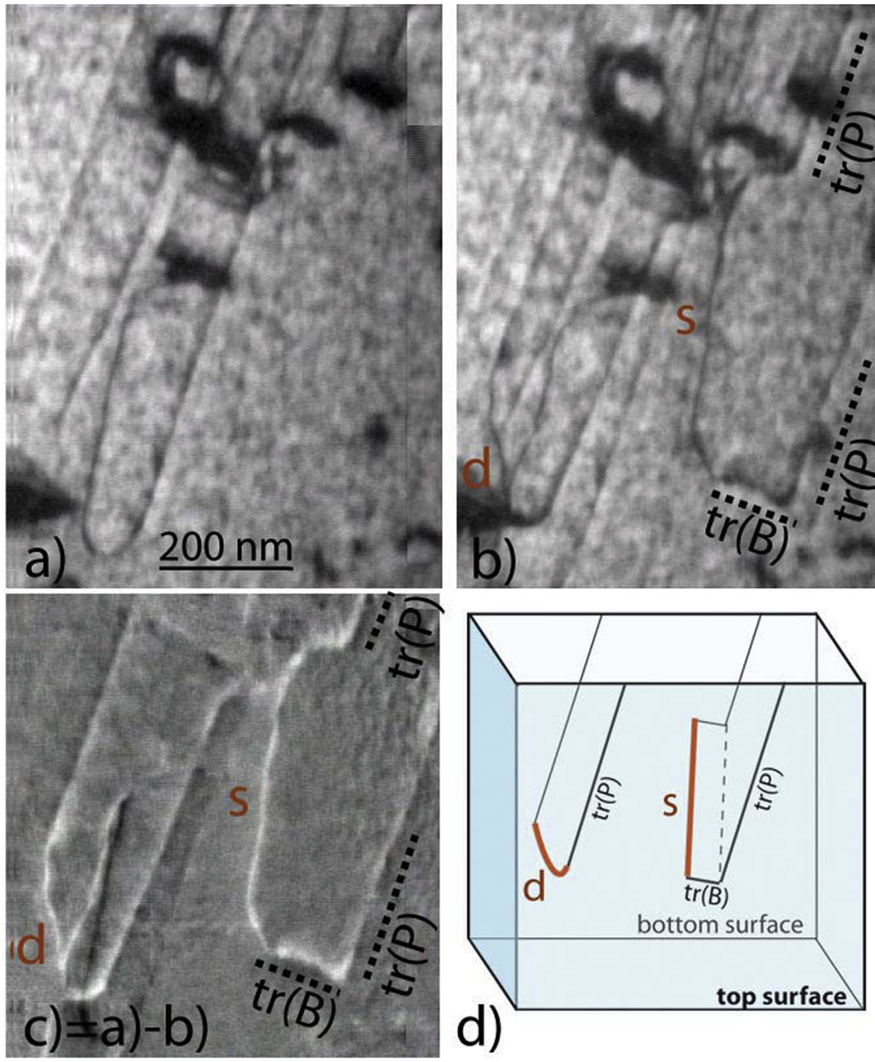

Fig. 4. Dislocations in Zr at 300 K. The curved dislocation d glides in its prismatic plane whereas the straight screw s glides in its prismatic plane (trace tr(P)) and cross-slips in the basal plane (trace tr(B)). Note the wavy aspect of tr(B). See video as a supplementary material.

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The above results show that the dislocation motion between 300 K and 573 K involves a mixture of prismatic and basal slip, for SF(B)/SF(P) = 1.36. Pyramidal slip may also be activated for SF(π)/SF(P) = 1.5, but this is difficult to prove unambiguously.

Fig. 8 shows curved dislocations gliding at 673 K in a prismatic plane with a high Schmid factor (SF(P) = 0.49, see Table 2). This grain exhibits a low basal Schmid factor (SF(B)/SF(P) ~ 0.08) and accordingly no basal slip is observed. On the other hand, two symmetrical pyramidal slip systems are highly stressed (SF(π)/SF(P) = 0.88 and 0.84). If pyramidal slip was easy, one would expect at least several cross-slip events, or at most wavy slip traces of directions in the acute angle defined by the two traces tr.π. Since the observed traces are perfectly rectilinear and parallel to tr.P, we can conclude that pyramidal slip in pure Zr is significantly more difficult than prismatic slip, even at high temperature.

Supplementary video related to this article can be found at https://doi.org/10.1016/j.actamat.2018.05.038

Similar results can be deduced from *in situ* experiments at high temperature on a commercial Zr alloy (this alloy described in Ref. [33] contains 1% Nb, 1500 ppm O, 370 ppm Fe and 40 ppm of Cr, in weight and is often referred to as Zr-1%Nb-O alloy): i) extensive basal slip of straight screw dislocations when the Schmid factor on the basal plane is more than twice that on the prismatic plane (SF(B)/SF(P) > 2) (see Table 2, unpublished), and ii) no pyramidal slip under moderately favorable stress conditions (SF(π)/SF(P) = 0.81 and 0.92, see Table 2, Fig. 7 of reference [33]).

On the other hand, *in situ* experiments carried out on a recrystallized Zircaloy-4 at room temperature show planar pyramidal slip for a particularly favorable orientation of the straining axis (SF(π)/SF(P) = 3.8) (Fig. 9). Straight screw dislocations move viscously, by double kink nucleation and propagation, against a high Peierls force, as in Ti at a lower temperature [18]. Pyramidal slip as an elementary system is thus certainly possible, but rather difficult in Zr.

3.2. Titanium

Experiments below room temperature on pure Ti have already been partly described in Ref. [18] and are first recalled. Then the new results obtained at room temperature and above are more extensively described.

Below room temperature, two different dislocation behaviors are observed, depending on the sample orientation and

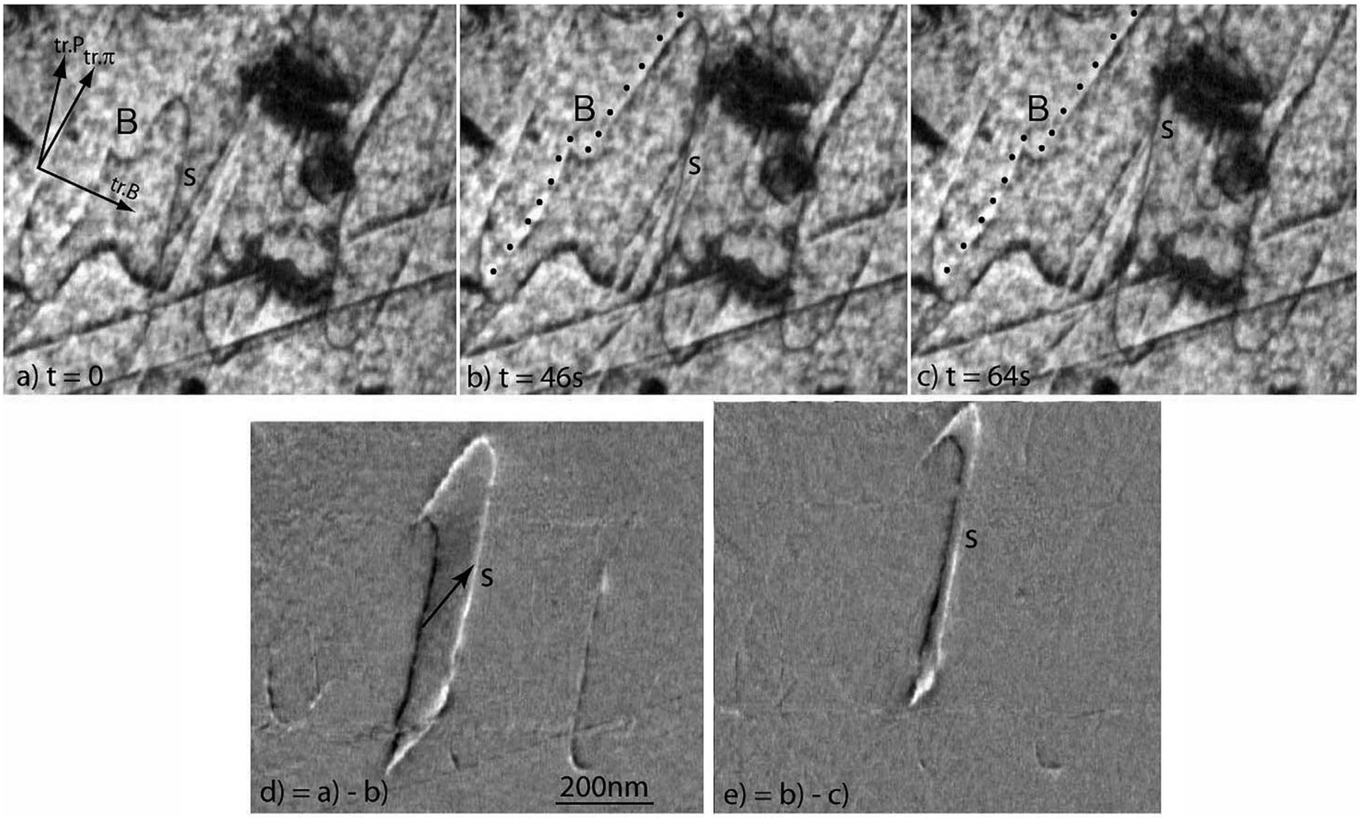

Fig. 5. Dislocations in Zr at 300 K. Same experiment as in Fig. 4. A straight screw dislocation noted s trails a wavy slip trace underlined by dots. This trace is almost parallel to the trace of a pyramidal plane (noted tr π), except for a small segment parallel to the trace of the basal plane (noted tr B), near the fixed point B. See video as a supplementary material.

corresponding Schmid factors. When the Schmid factor of the most activated pyramidal plane is close to, or smaller than that of the prismatic plane, dislocations exhibit a planar motion in the prismatic plane, as in zirconium in the same temperature range. As shown in Fig. 2 of reference [18], dislocations gliding in this plane are curved and do not show any tendency to straighten along their screw direction, which indicates an easy motion and negligible Peierls friction forces. When one pyramidal cross-slip system is at least 1.1 times more stressed than the prismatic one, prismatic-pyramidal cross-slip becomes possible, which results in two different situations depending on resolved shear-stress values. For high values of SF(π)/SF(P) (~1.7 in the case of Fig. 1 of [18], see Table 2), straight screw dislocations can glide slowly and viscously in the pyramidal plane, as in Zr under similar conditions (Fig. 9), until the reverse cross-slip brings the dislocation back in the prismatic plane. For smaller values of SF(π)/SF(P) between 1.1 and 1.7, cross-slip is just a change of core configuration with no subsequent motion. Then, dislocations are only blocked in their screw direction in the prismatic plane until they are released by the reverse change of core configuration. This latter behavior, also called locking-unlocking, has been observed only in Ti (Fig. 4 of [13]).

At room temperature, prismatic slip remains the only observed slip system in samples where pyramidal slip and basal slip are only moderately stressed (i.e. Schmid factors less than 1.5 times that of prismatic slip, see Table 2). Under such conditions, curved dislocations glide easily and trail rectilinear slip traces parallel to the intersection between the prismatic plane and the foil surface, as shown in Fig. 10.

Supplementary video related to this article can be found at https://doi.org/10.1016/j.actamat.2018.05.038

No straight screw dislocation is observed at room temperature, unless basal slip is sufficiently stressed. Fig. 11 shows a single-arm dislocation source rotating anti-clockwise in a sample where both the basal plane and one pyramidal plane are two-times more stressed than the prismatic plane (SF(B)/SF(P) = 2, SF(π)/SF(P) = 2.3, Table 2). The rotating dislocation clearly exhibits a straight part along the screw direction, typical of a high Peierls friction force. The slip traces at the two foil surfaces are in average parallel to the trace of a pyramidal plane. A component of pyramidal slip would not be surprising, since this system has already been observed under similar sample orientations and at a lower temperature (see above). However, the slip trace directions are locally clearly out of the acute angle defined by the traces of prismatic and pyramidal planes, which shows that, according to Fig. 1, basal slip is necessarily involved. This conclusion is supported by Fig. 12 showing another source in the same sample. This source also rotates anti-clockwise and involves straight screw dislocations. The slip traces (underlined by dots) are now almost parallel to the intersection of the basal plane and the foil surface, showing that basal slip is clearly activated.

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4. Discussion

4.1. Slip systems in Zr and Ti

The experimental results show that Zr and Ti have many common properties (easy glide in prismatic planes, cross-slip into the basal plane above room temperature), but also some differences, especially concerning the activation of pyramidal slip. These analogies and differences are discussed below.

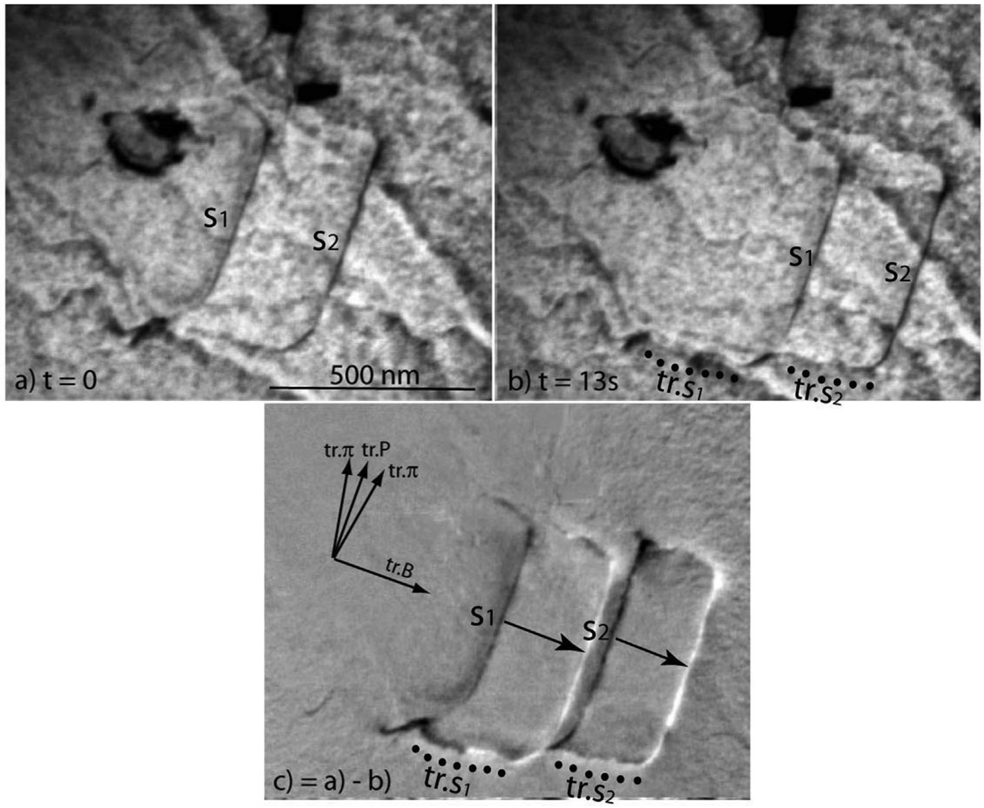

Fig. 6. Screw dislocations s_1 and s_2 gliding in a wavy surface of Zr strained at 373 K. The slip traces underlined by dots and noted tr(s_1) and tr(s_2) are wavy but close to that of the basal plane (trB). See video as a supplementary material.

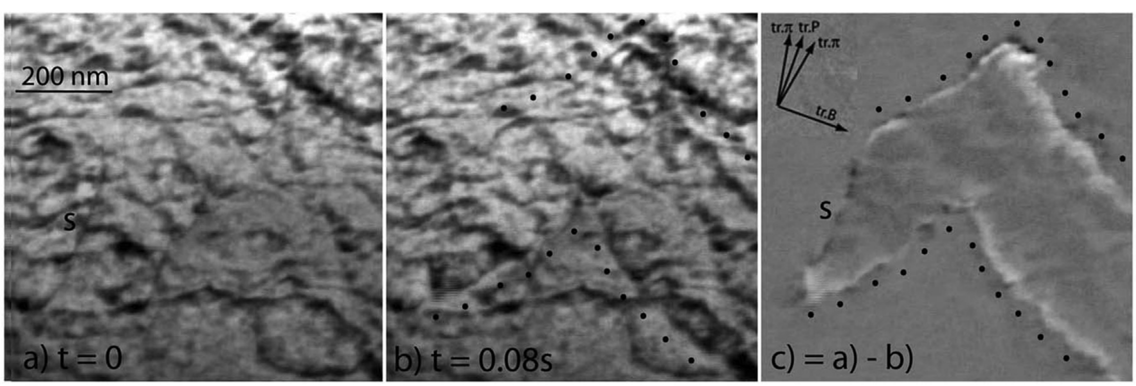

Fig. 7. A screw dislocation s visible in (a), glides rapidly in (b) in a wavy surface of Zr strained at 573 K. The two slip traces on the two foil surfaces are underlined by dots. Their average direction is close to the trace of the basal plane (trB). See video as a supplementary material.

4.1.1. Slip systems in Zr

The most interesting new results in Zr are the observation of basal slip at and above room temperature, the observation of pyramidal slip for especially favorable stress conditions, and the analysis of the stress conditions for observing the different slip systems. Results on prismatic slip already presented in Ref. [18] are recalled in order to give a complete overview of all slip systems.

Prismatic slip is the only observed system below room temperature, for any orientation of the applied stress (except parallel to the **c**-axis). Cross-slip becomes important only at and above 300 K, and slip trace analysis shows that it involves at least prismatic slip and basal slip. This agrees with the strong correlation between cross-slip activity and stress in the basal plane (intensive cross-slip for the Schmid factor ratio SF(B)/SF(P) = 1.2, no cross-slip for SF(B)/SF(P) = 0). Basal slip is thus considered to be slightly more difficult than prismatic slip, at and above room temperature.

Cross-slip into the pyramidal plane may also occur at and above 300 K because observations of wavy traces exhibit some correlation with the stress intensity in the most activated pyramidal plane (intensive cross-slip for SF(π)/SF(P) = 1.5, no cross-slip for SF(π)/SF(P) = 0.9). However, since pyramidal slip has been unambiguously isolated only for very specific stress conditions (SF(π)/SF(P) = 3.8) in a Zr alloy, and since it has been excluded at high temperature for ordinary stress conditions (SF(π)/SF(P) = 0.8 to 0.9), in pure Zr and in a Zr alloy, its exact importance as a function of stress remains a subject of controversy.

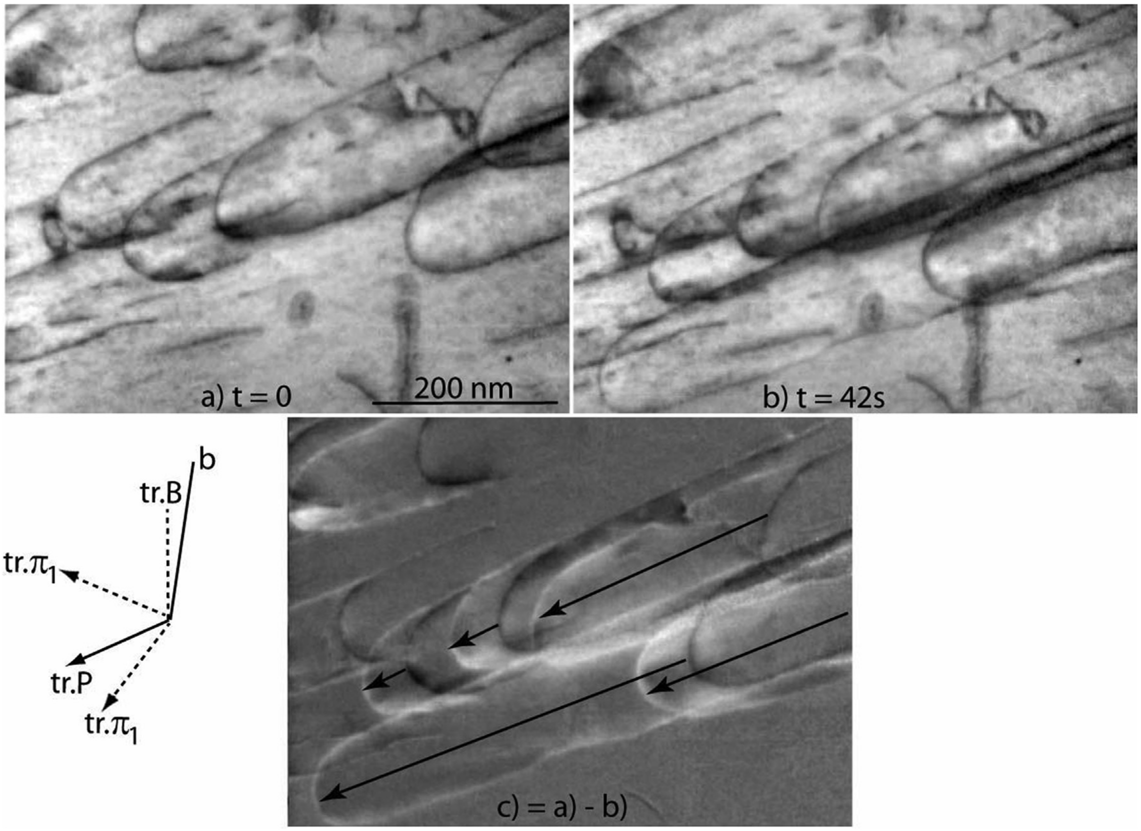

Fig. 8. Dislocations gliding in a prismatic plane of Zr strained at 673 K. b denotes the projection of the Burgers vector direction. No cross-slip is observed in any plane. See video as a supplementary material.

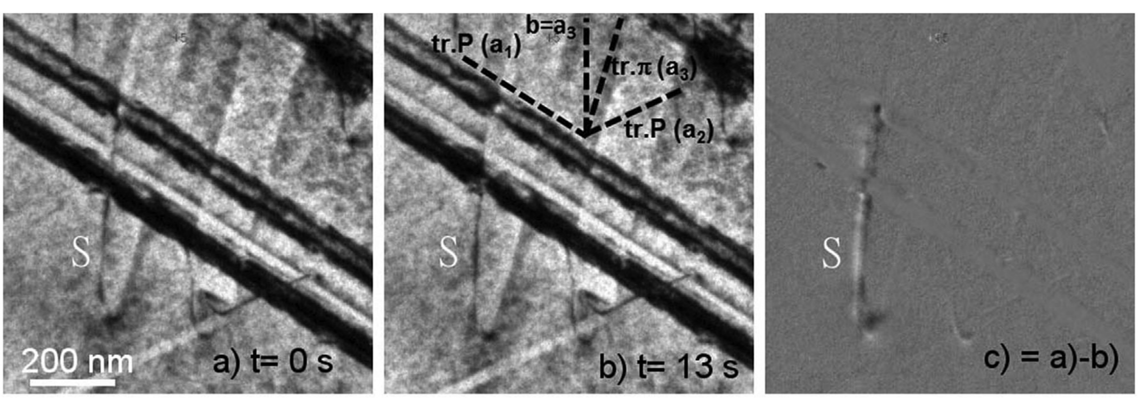

Fig. 9. A straight screw dislocation noted s with Burgers vector a_3 glides slowly and viscously between (a) and (b), in a recrystallized Zy-4 alloy strained at 300 K. The motion can be seen in the image difference (c). The projection of the Burgers vector a_3 and the orientation of the slip traces are given on (b).The slip traces tr π (a_3) are parallel to those of the pyramidal plane containing the Burgers vector a_3. In the same grain, two prismatic slip systems P(a_1) and P(a_2) corresponding to the two other <a> Burgers vectors, are also activated.

4.1.2. Slip systems in Ti

The most interesting new results in Ti are the observation of basal slip at and above room temperature, and the analysis of the stress conditions for observing the different slip systems. Some results on prismatic and pyramidal slip already presented in Ref. [18] are also recalled.

Below room temperature prismatic slip is the main glide system in Ti. However, contrary to what has been observed in Zr, dislocations can cross slip onto a pyramidal plane for a sufficiently high resolved shear stress, at 150 K. This is supported by observations of cross-slip and pyramidal slip for SF(π)/SF(P) = 1.7 published in Ref. [18], and by new observations of (i) cross-slip but no detectable pyramidal slip for SF(π)/SF(P) = 1.1, and (ii) no cross-slip at all in another sample for SF(π)/SF(P) = 1.2. Pyramidal slip is thus considered to be about ~1.5 times harder than prismatic slip at 150 K. It is not observed at 300 K for SF(π)/SF(P) = 1.2 (and SF(B)/SF(P) = 0), which shows that it remains more difficult than prismatic slip.

The slip traces are made of straight segments parallel to the intersections of prismatic and pyramidal planes with the foil

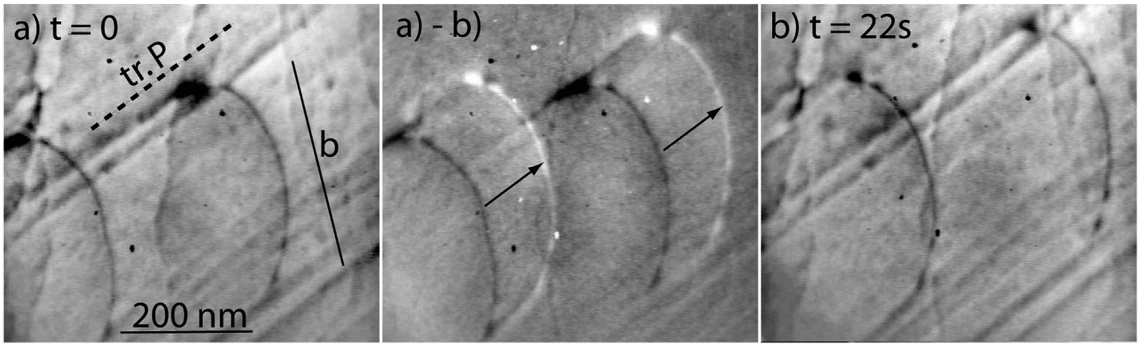

Fig. 10. Dislocations gliding in a prismatic plane of Ti strained at 300 K (slip trace Tr.P). b denotes the projection of the Burgers vector direction. No cross-slip is observed in any plane. See video as a supplementary material.

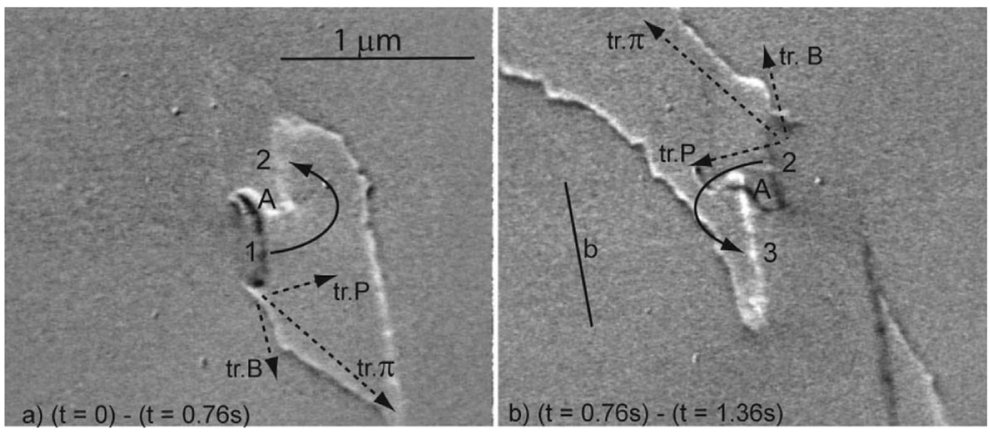

Fig. 11. Difference-images showing the rotation of a dislocation source in Ti strained at 300 K. The straight screw segment (direction b) rotates around the pinning point A, from 1 to 2 in (a), and from 2 to 3 in (b). The wavy slip traces on the two sample surfaces are in average parallel to tr.π in (b), but exhibit many segments parallel to tr.B in (a) and (b). See video as a supplementary material.

surface. This shows that pyramidal slip is an elementary slip system in Ti, namely that it is not the result of intensive cross-slip between other slip systems.

At 300 K, cross-slip is very intensive when available cross-slip planes are sufficiently stressed. Slip trace analysis shows that cross-slip involves at least prismatic and basal slip. Pyramidal slip which should be easy at room temperature is also probably involved, although this cannot be proved unambiguously in our experiments. The occurrence of cross-slip into basal and presumably pyramidal planes is supported by the analysis of the corresponding Schmid factors, showing that cross-slip takes place for SF(B)/SF(P) = 2 and SF(π)/SF(P) = 2.3, not for SF(B)/SF(P) = 0.5 and 0.7 and SF(π)/SF(P) = 1 and 1.2.

It can thus be concluded that $0.7 < \tau_B/\tau_P < 2$ in Ti, namely that basal slip is probably slightly more difficult than prismatic slip at and above room temperature. This result is in qualitative agreement with the pure shear experiments conducted by Sakai and Fine [15] and Levine [8] showing that the critical resolved shear stress of basal slip in Ti and Ti-5.2%Al is two to three times that of prismatic slip, at and above room temperature. It is also in satisfying agreement with the results of [19], and with the values of τ_B/τ_P discussed in the introduction, ranging between 1.1 and 2.1 [24–27].

4.2. Properties of the different slip systems in Zr and Ti

4.2.1. Prismatic slip

In the absence of any cross-slip, prismatic slip in pure Zr and Ti consists of curved dislocations gliding easily, except for some weak pinning points. Since the density of these pinning points appears to be higher in a Zr alloy than in pure Zr (Van Arkel and sponge), they can be attributed to solute atoms, in particular oxygen. This is in agreement with results in Ref. [19], and with atomistic calculations showing that dislocation cores tend to spread in the prismatic plane of Ti and Zr [17,34,35] and that the corresponding Peierls frictions force is actually very weak in Zr [17,36].

We thus conclude that prismatic slip alone is controlled by the elastic interaction between dislocations and solute oxygen atoms, not by a Peierls friction force. Since prismatic slip is activated for any crystal orientation (except when the stress is applied along the **c**-axis), this accounts for the strong macroscopic hardening due to oxygen, as discussed in the introduction. The increase of yield stress with decreasing temperature is easily explained by the thermally activated crossing of fixed small-size obstacles (oxygen atoms) by dislocations (see e.g. chapter 3 of reference [37]).

Straight screw dislocations moving slowly and steadily are the result of cross-slip in either pyramidal or basal planes, only for favorable crystal orientations enhancing those cross-slip systems. Under such conditions, the hypothesis of a Peierls friction force controlling prismatic slip is a misinterpretation of the role of straight screw dislocations when cross-slip is activated. Indeed, if we return to the observations of straight screw dislocations in Zr [4,16], Ti [12–14], and Ti-5.2%Al [15], we find that they all correspond to situations where cross-slip in either pyramidal or basal planes can be activated.

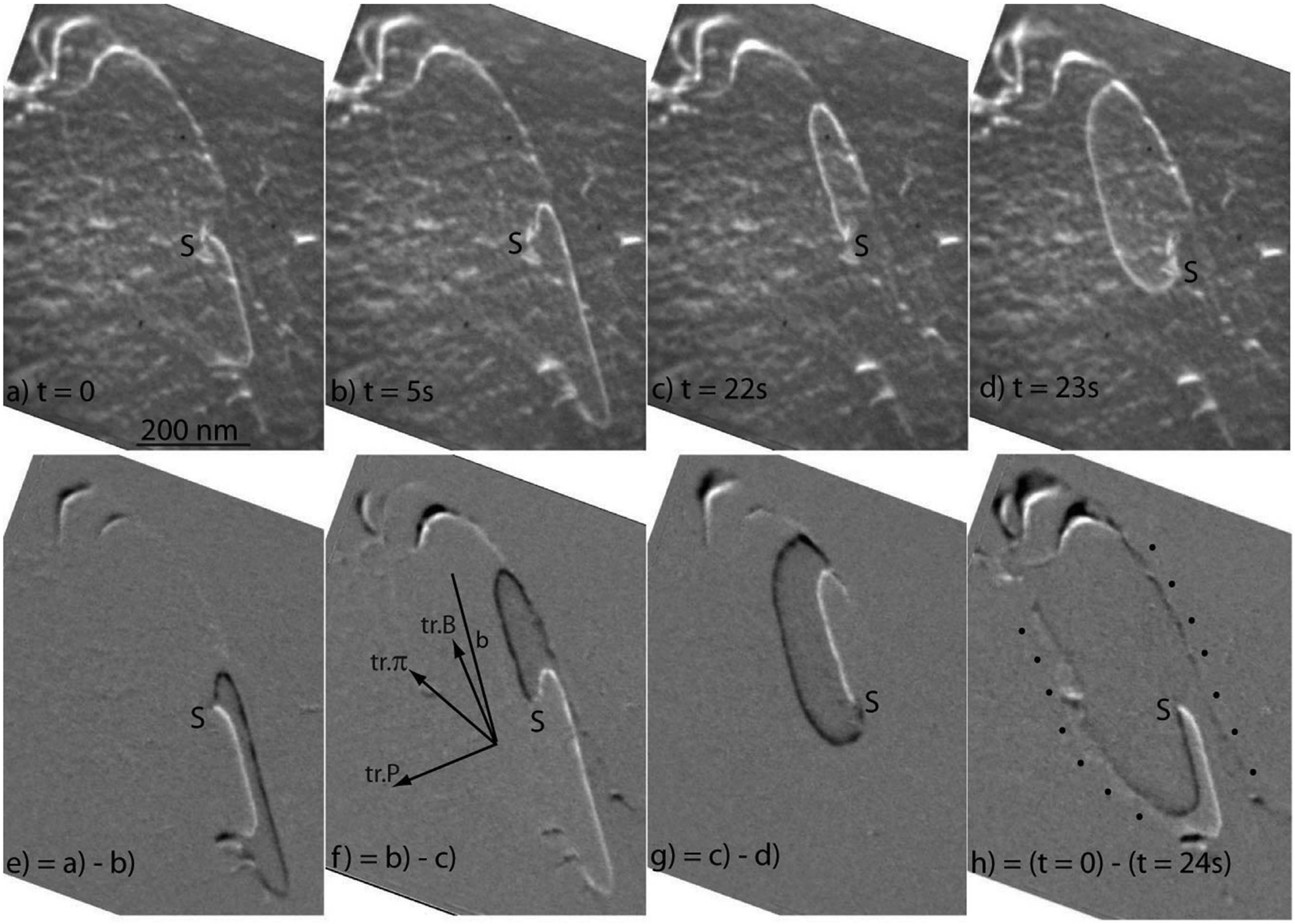

Fig. 12. Dislocation source in Ti strained at 300 K. The straight screw segment (direction b) rotates anti-clockwise and trails wavy slip traces underlines by dots. The slip trace direction is very close to tr.B. See video as a supplementary material.

Locking by cross slip into the pyramidal plane at low temperature in Ti induces a jerky prismatic slip, which probably contributes to decrease the corresponding average dislocation velocity. Consequences for the mechanical properties will be discussed in section 4–3 below. This does not occur in Zr, probably as a result of a higher ratio of the core energies of screw dislocations in the pyramidal and prismatic planes [18].

4.2.2. First order pyramidal slip of ***a***-dislocations

According to section 4–1, pyramidal slip is considered to be definitely harder than prismatic slip at all temperatures. Estimated ratios are about 1.5 in Ti at low temperature, and of the order of 1.5–3 at and above room temperature in Zr.

When activated, this slip system is subjected to a strong Peierls friction, in agreement with computations in Ref. [18]. It proceeds by the kink pair mechanism as shown by the straight screw dislocations moving slowly. This friction force is probably at the origin of the high critical resolved shear stress estimated above.

4.2.3. Basal slip

Basal slip has similar properties in Zr and Ti. It becomes intensive at and above room temperature but probably remains slightly more difficult than prismatic slip. It is characterized by straight screw dislocations moving viscously in wavy surfaces, and by curved non-screw parts moving much faster.

These features are typical of a Peierls mechanism where screw dislocations glide across Peierls valleys by a kink-pair mechanism. In the present case, screw dislocations have their lowest energy when their core is extended either in the prismatic (Zr, Ti) or in the pyramidal (Ti) planes [17,18,34,35]. Under such conditions, glide in the basal plane proceeds by series of kink-pair nucleations schematized in Fig. 13. A wavy motion is expected if screw dislocations are extended in their prismatic plane where they are very mobile at each atomic row, and a planar motion is expected if they are extended in a pyramidal plane where they are much less mobile. Since the motion is clearly wavy, we can conclude that screws are extended in the prismatic plane, namely that the wavy motion is a combination of prismatic and basal slip. This situation is similar to that observed in magnesium deformed in prismatic slip at room temperature, provided the roles of prismatic and basal slips are reversed [38].

Basal slip is clearly an elementary system, because it is not a combination of other slip systems, and because it is activated by the shear stress in the basal plane. It is however never planar since it is always combined with prismatic slip.

4.3. Consequences for some aspects of the mechanical behavior

Since both Ti and Zr deform by combinations of different slip systems controlled by different mechanisms, their mechanical

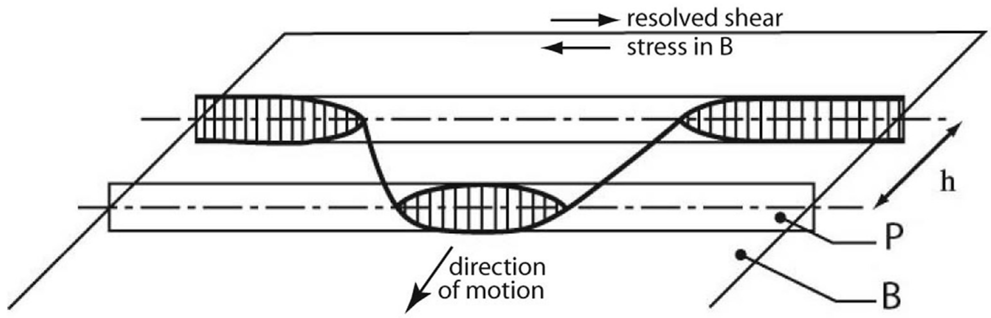

Fig. 13. Schematic description of the kink-pair mechanism in the basal plane. The core extended in the prismatic plane remains highly mobile in this plane at each atomic row, which accounts for the observed wavy motion.

properties are necessarily complex. However, a few clear conclusions can be derived.

Below room temperature, Zr deforms by pure prismatic slip controlled by the interaction between mobile dislocations and fixed oxygen atoms. This accounts for the strong dependence of yield stress and activation volume on oxygen content discussed in details in Refs. [1–7]. In the same low-temperature range, Ti deforms by a mixture of prismatic slip controlled by the same interactions with oxygen atoms, and locking by cross-slip into the pyramidal planes followed by unlocking by the reverse cross-slip [13,18]. The first mechanism accounts for the strong similitude of Zr and Ti, in particular the dependence of yield stress and activation volume on oxygen concentration. However, for very low oxygen concentrations, activation volumes vary as the inverse of the stress squared in Ti, which cannot be accounted for by the effect of oxygen atoms [31]. Then, the unlocking of screw dislocations by pyramidal-prismatic cross-slip may be rate-controlling, as proposed in Ref. [18].

At room temperature, Zr and Ti exhibit the same discontinuity characterized by a peak in the activation area versus temperature curve [3,5,8,12], and by a small hump in the corresponding stress versus temperature curve. In Zr, this corresponds to the end of planar prismatic slip and to the activation of cross-slip onto the basal plane. In Ti where cross-slip is already possible onto the pyramidal plane, this also corresponds to the onset of cross-slip onto the basal plane. Under such conditions, the peak of activation area can be interpreted in two different ways:

i) Following references [12,14,28], the onset of cross-slip at 300 K can induce a substantial strain-hardening at the origin of the hump and associated peak. This interpretation is plausible provided the prismatic-pyramidal cross-slip of the original formulation is replaced by the prismatic-basal one.
ii) Referring to the pure shear experiments of Levine in Ti [8], the increase of activation area between 250 K and 300 K can be interpreted by prismatic slip becoming athermal, and the decrease of activation area above 300 K by a transition towards the activation area of pure basal slip (Fig. 14). However, according to this second interpretation, the activation area above 300 K should correspond to the kink-pair mechanism described in section 4-2-3, and be independent of the oxygen content. Since it is clearly not the case, the strong interaction between screw dislocations and oxygen atoms calculated in Ref. [39] should also be taken into account.

5. Conclusions

In situ straining experiments inside a TEM have yielded important results concerning the elementary slip systems involving **a**-type dislocations in zirconium and titanium.

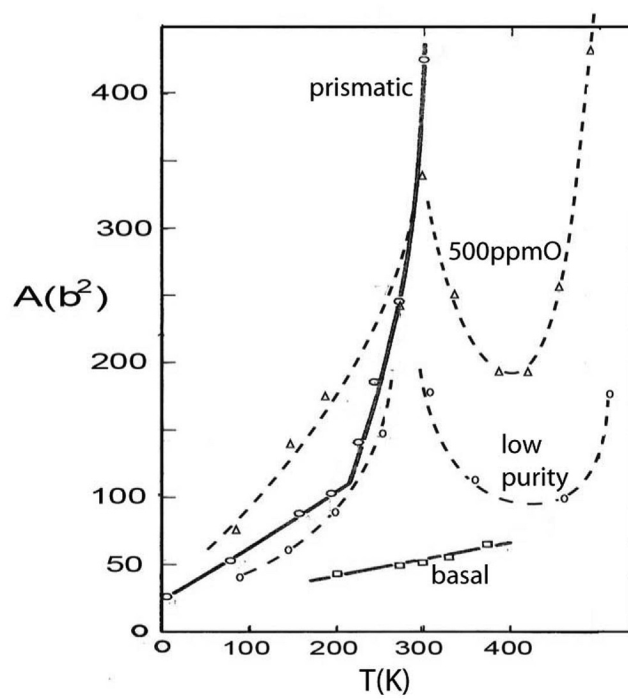

Fig. 14. Comparison between the activation area of Ti measured in pure shear in prismatic and basal planes by Levine (full line [8]), and in tensile deformation by Akhtar and Teghtsoonian (two different purities, dotted lines [5]).

- Below room temperature, prismatic slip is controlled by the elastic interaction between mobile dislocations and small-size obstacles, presumably oxygen atoms. Peierls friction forces are negligible, in agreement with recent ab-initio calculations in Zr.
- Dislocation locking by prismatic-pyramidal cross-slip is easier in Ti than in Zr, owing to different ratios of core energies discussed in Ref. [18].
- Pyramidal slip is a rather difficult process in both Ti and Zr, which requires favorable stress conditions (SF(π) definitely larger than SF(P)). It is controlled by a high Peierls friction force along the screw direction, in agreement with ab initio calculations [18].
- Glide in the basal plane becomes important above room temperature in Zr and Ti, provided the basal plane is sufficiently stressed (SF(B) at least equal to SF(P)). Basal slip involves the slow and viscous motion of straight screw dislocations, in agreement with a kink-pair mechanism where screw dislocations are extended in the prismatic plane. Although wavy and mixed with prismatic slip, it is clearly an elementary slip system.

- Except for an easier locking by prismatic-pyramidal cross-slip in Ti at low temperature, Zr and Ti exhibit close dislocation properties in the same temperature ranges. This accounts for several common mechanical properties including the strong influence of oxygen content and the discontinuous behavior at room temperature.

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