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Benjamin Galy, Michael Musi, Muriel Hantcherli, Guy Molénat, Alain Couret, et al.. Glide and mixed climb dislocation velocity in γ -TiAl investigated by in-situ transmission electron microscopy. Scripta Materialia, 2023, 228, pp.115333. 10.1016/j.scriptamat.2023.115333 . hal-04281588

HAL Id: hal-04281588

<https://hal.science/hal-04281588>

Submitted on 13 Nov 2023

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**Glide and mixed climb dislocation velocity in γ -TiAl
investigated by in-situ transmission electron microscopy**

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Abstract

Dislocation velocities at high temperatures in metallic systems are believed in literature to be different for glide and climb, the values being bigger in case of glide. However, this has not been experimentally established. Therefore, in this study, dislocation velocities were measured with simultaneous determination of the corresponding mechanism (glide or mixed climb). For this purpose, coupled experiments of measurements of dislocation velocities by in-situ TEM investigations and of determination of movement planes by stereographic analyses have been carried out at 770-790°C in the γ phase of an intermetallic Ti-48.4Al-0.1B (at.%) alloy. Mixed climb and pure glide mechanisms have thus been identified, both leading to dislocation velocities in the same order of magnitude (in the 0.5-5 nm/s range), showing that within a transition temperature domain, mixed climb can reach the velocity of glide.

Keywords: transmission electron microscopy (TEM); titanium aluminides; dislocation mobility; in-situ TEM.

Dislocation climb/glide is among the classical mechanisms proposed to account for the deformation of metallic systems at high temperatures [1,2]. In this description, the motion of the dislocations is supposed to involve glide and climb contributions, the deformation rate being controlled by the slower of the two. In these classical theories, the climb velocity v_c is assumed to be less than the glide velocity v_g , because climb requires diffusion of vacancies, which is considered a slow process under creep conditions. However, in our knowledge, the assumption that gliding and climbing dislocations exhibit different velocities has not been experimentally established, probably because the velocity of the dislocations and the

deformation mechanism (glide or climb) are difficult to determine simultaneously. In particular, determining the mechanism requires establishing the movement plane of the dislocations, which can only be performed by stereographic analyses. Therefore, we used for the first time in-situ TEM experiments coupled with stereographic analyses to measure dislocation velocities and, at the same time, to determine the movement planes of these dislocations. Investigations of deformation mechanisms at high temperatures are crucial for developing materials, which are able to endure thermo-mechanical loading as, e.g., experienced by hot parts used in aeronautical engines. In this context, intermetallic γ -TiAl based alloys are interesting examples, as they are already introduced in engines of aircrafts used in commercial flights [3].

At high temperature, dislocation motion occurs in planes referred to as movement planes P . Recall that glide and climb lead to the Burgers vector \vec{b} being contained in P or perpendicular to P , respectively. In intermediate situations (\vec{b} making any angle different from 0° and from 90° with P), the deformation mechanism is the motion of jogged dislocations [4]. As it involves climb and glide components, it is sometimes called mixed climb [5,6], a terminology which will be kept here.

The deformation in the TiAl alloys is often concentrated on the γ phase, which is of ordered tetragonal $L1_0$ crystal structure. Its main mode involves two families of ordinary dislocations of Burgers vectors $a/2 [110]$ and $a/2 [1\bar{1}0]$. At low temperatures (20°C to *ca.* 400°C), deformation occurs by glide of elongated screw segments in $\{111\}$ planes controlled by Peierls-type periodic frictional forces resulting in jumps between sessile positions (locking-unlocking mechanism) with superimposition of anchoring effects on pinning points [7-9]. At higher temperatures (400°C to 600°C), the deformation involves bursts of numerous screw dislocations, corresponding to dynamic strain ageing [10,11]. At 550°C to 850°C , climb is activated [6,7,12,13]. From 900°C and above, other mechanisms are reported: glide of ordinary dislocations in non-compact planes [14], increased activation of superdislocations [15] as well as formation of new c-type $a[001]$ dislocations [16]. However, in none of the above studies, the question of the respective velocities of the gliding and climbing dislocations is addressed. Therefore, in the present work, in-situ TEM experiments were carried out with the aim to simultaneously measure dislocation velocities and to determine the corresponding mechanism, in particular to establish the movement planes.

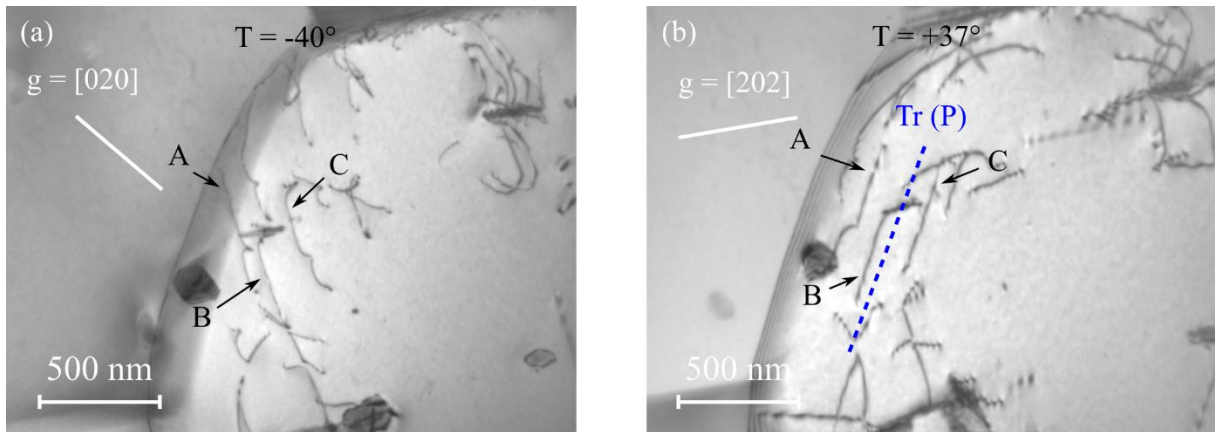
For this purpose, a binary Ti-48.4Al-0.1B (at.%) alloy was selected as model system, which was elaborated at 1300°C in a near- γ microstructure by spark plasma sintering (SPS) from prealloyed powder (GfE Metalle und Materialien GmbH, Nuremberg, Germany), employing standard cycles and sample temperature calibrations [17]. The oxygen content of the powder was 573 mass ppm. Cylindrical samples of 36 mm in diameter and 8 mm in thickness were obtained, from which tensile samples with 2.2 mm in gauge diameter were machined. Creep tests at 800°C under a constant tensile load σ of 36 MPa were conducted in air employing an Adamel machine, using a methodology described previously [13,18]. The resulting minimum creep rate was $3 \times 10^{-7} \text{ s}^{-1}$ and the tests were interrupted after 3% of

plastic strain. Rectangular TEM thin foils (0.8 mm × 3 mm) were extracted with their long axis parallel to the tensile axis and prepared by electro-polishing. The in-situ TEM experiments consisted of heating the TEM thin foils in a Gatan heating double tilt sample holder. Due to thermal expansion of the thin foils, which are constrained in the sample holder, compressive stresses are generated locally, yielding movement of pre-existing dislocations and, more rarely, inducing nucleation of new ones. Due to the high temperatures reached in these experiments (700-900°C), degradation of the thin parts of the samples by oxidation occurred in 2 to 5 minutes, leading to dramatic contrast loss, thus allowing only little time for the observations. To allow post-mortem observations, the in-situ experiments were interrupted before the degradation of the thin foil reached too high levels. The thin foils were then removed from the heating sample holder and positioned in a Gatan tilt-rotation sample holder. The microscope used was a JEOL 2010 TEM.

In this first part, the specimens are studied at room temperature by post-mortem TEM observations, after deformation by creep at 800°C. Figures 1 and 2 show examples of the determination of glide and mixed climb mechanisms, respectively, based on the determination of respective orientations of Burgers vector \vec{b} and of movement plane P from post-mortem TEM observations. The occurring Burgers vectors \vec{b} have been determined using the invisibility criterion ($\vec{g} \cdot \vec{b} = 0$, with \vec{g} being the diffraction vector) by standard procedure not detailed here. The orientations of the movement planes P have been obtained by observation of curved dislocations at different inclinations of the sample, supposing that the curvature of the dislocation results from its motion in the movement plane. Therefore, determining the plane defined by the curved dislocation line can be achieved by two methods. The first method consists of inclining the sample until the dislocation line is seen as a rectilinear segment (see Fig. 1), meaning that the movement plane is edge-on. However, the possibility to employ this method is not frequent. Therefore, another method can be used, consisting in aligning the two emergence points of a curved dislocation at the surface of the thin foil parallel to the tilt axis of the sample holder, using a tilt-rotation sample holder. The projected width w between characteristic points on the dislocation line (middle of the line, geometrical singularity, etc.) and the line joining the emergence points is then measured on the micrographs for various tilt angles, for which the dislocation is in contrast. The dependence of w in function of the tilt angle T is:

$$w = w_0 \cos(T - T_0), \quad (1)$$

where w_0 is the maximum of the projected width, obtained when the movement plane is horizontal, and T_0 is the angle between the movement plane and the thin foil plane. Then, this relation is fitted to the measurements of w , which allows determining w_0 and T_0 . The requirement for this method to be valid is that the two extremities of the dislocation line emerge on the same side of the thin foil. Figure 2 shows an example of the determination of the movement plane in the case of mixed climb using this method. Considering the various sources of experimental errors, the accuracy of the orientations is of the order of $\pm 5^\circ$.



(c) Stereographic projection for $T = +37^\circ$

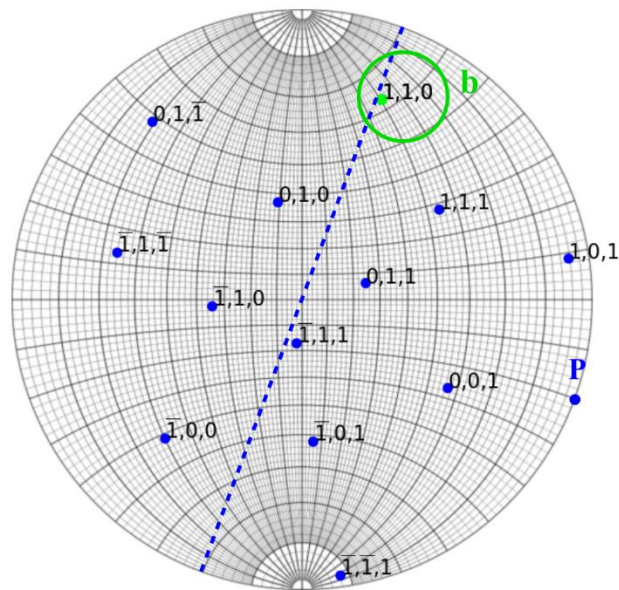


Figure 1. (a-b) TEM micrographs of three dislocation segments, labelled A, B and C, for two inclinations T of the sample holder. In micrograph (b) the dislocations are rectilinear, indicating that the movement plane is edge-on (blue dashed line). (c) Stereographic projection of the sample inclined by $+37^\circ$, showing that the movement plane P of the dislocations (blue dashed line, and pole P) contains the Burgers vector $\vec{b} = a/2 [110]$ (circled in green), which indicates a glide mechanism. This plane does not correspond to low indices crystallographic planes.

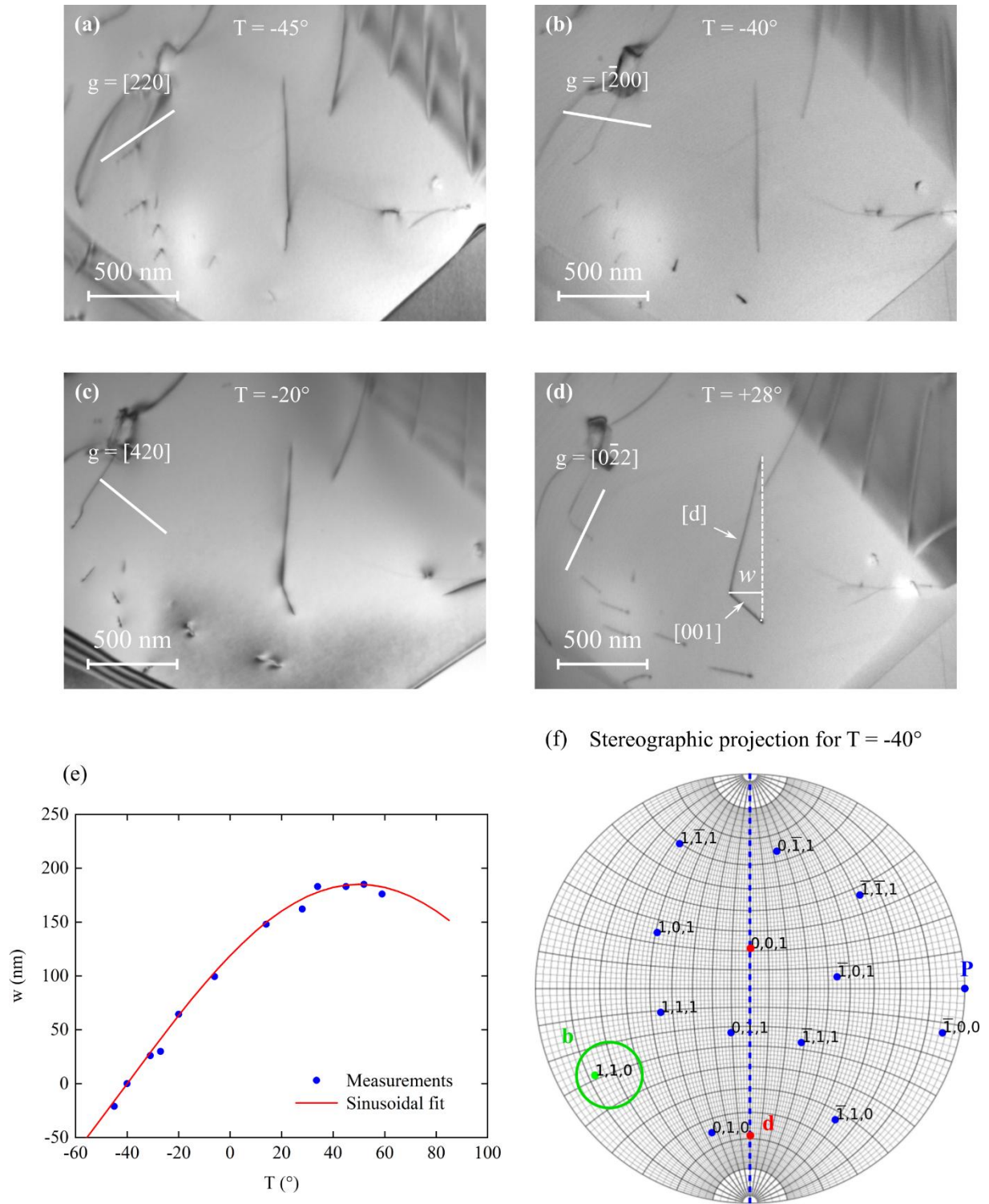


Figure 2. (a-d) TEM micrographs of a dislocation segment of angular morphology for different inclinations of the sample. (e) Graph of the projected width w as a function of the tilt angle of the sample holder T , showing that $w = 0$ (movement plane edge-on) for $T = -40^\circ$, a situation which is visible in micrograph (b). (f) Stereographic projection of the sample inclined by -40° , showing that the movement plane P of the dislocations (blue dashed line, and pole P) does not contain the Burgers vector $\vec{b} = a/2 [110]$ (circled in green), which

indicates a mixed climb mechanism. This plane is close to the $(\bar{1}00)$ plane. Directions $[001]$ and $[d]$ are indicated by red dots.

Therefore, Fig. 1 shows that the Burgers vector is contained in the movement plane, whereas in Fig. 2 it is not. The first case thus shows glide, which occurs in a plane not belonging to the $\{111\}$ family and corresponds then to cross slip. In the second case, the dislocation moved by mixed climb. This dislocation exhibits an angular morphology with one segment aligned along $[001]$, the second referred to as $[d]$ being not far from the $[010]$ direction. Overall, ten tilt experiments have been performed, but because the emergence points of the dislocations were situated on the same side of the thin foil about one in two times, only four experiments were valid. Among them, two observations corresponded to glide and two to climb. This shows that glide and climb coexist during creep at 800°C in the γ phase of the investigated binary TiAl alloy, as also reported previously for a TiAl alloy which was deformed during a creep test conducted at 850°C [13].

Furthermore, in the case of glide, knowing the orientation of the movement planes, the Schmid factor S_g can be obtained as follows:

$$S_g = \cos(\vec{T}, \vec{b}) \cdot \cos(\vec{T}, \vec{n}), \quad (2)$$

with \vec{T} being the vector along the tensile axis (vertical direction in the TEM micrographs), and \vec{n} representing the normal to the movement plane. Using S_g , the critical resolved shear stress τ_g can be obtained from Eq. 3:

$$\tau_g = S_g \cdot \sigma, \quad (3)$$

with σ indicating the tensile stress (= 36 MPa).

Similarly to the case of glide, in the case of climb [19], a projection factor called “climb factor” can be defined as $S_c = \cos(\vec{T}, \vec{b}) \cdot \cos(\vec{T}, \vec{n})$ and allows to calculate the resolved climb stress, given by $\tau_c = S_c \cdot \sigma$. Because the Burgers vector and the normal of the movement plane are not perpendicular to each other contrary to the case of glide, S_c is not limited to 0.5 and could vary between 0 and 1. Therefore, we give in Tab. 1 Schmid and climb factors as well as critical resolved shear and climb stresses, where it can be seen that the resolved stresses for both mechanisms are very close.

Table 1. Schmid factors S_g , climb factors S_c , critical resolved shear and climb stresses τ_g and τ_c determined from the data of the tilt experiments.

Mechanism	(\vec{T}, \vec{b}) ($^\circ$)	(\vec{T}, \vec{n}) ($^\circ$)	S_g, S_c	τ_g, τ_c (MPa)
Glide	33	60	0.42	15
Glide	33	66	0.34	12
Mixed climb	48	41	0.50	18
Mixed climb	53	60	0.30	11

Then, dislocation velocities have been measured during in-situ heating TEM experiments using video sequences and the movement planes of these dislocations have been determined by tilt experiments. Note that the true distances of the dislocation motion have been geometrically corrected using the known orientations of the movement planes with respect to the image plane. Finally, the measurement uncertainty of the dislocation velocity mainly depends on the precision of the positioning of the dislocation line, which was typically of ± 10 nm.

Figure 3 shows micrographs acquired during an in-situ experiment at 770°C evidencing pure glide. Figure 3a displays an image taken on the right part of a crystalline grain. Figures 3b-c display two images extracted from a video sequence in which the dislocation labelled A exhibits a continuous movement, different from the discontinuous movement by locking-unlocking observed at room temperature [9]. For this dislocation, a velocity of 1.2 ± 0.2 nm/s is measured. Figures 3d-e show the left part of the same crystalline grain upon reaching 770°C and after further 90 s of holding. Many dislocations have moved, as shown by the slip traces seen in Fig. 3e, allowing to measure a velocity of 4.4 ± 0.2 nm/s for the dislocation labelled B. In the following, the mean value, 2.8 nm/s, will be considered. Finally, Figs. 3a-c and 3d-e show that the dislocation lines are not elongated along the Burgers vector, which means that the dislocations possess mixed edge and screw character.

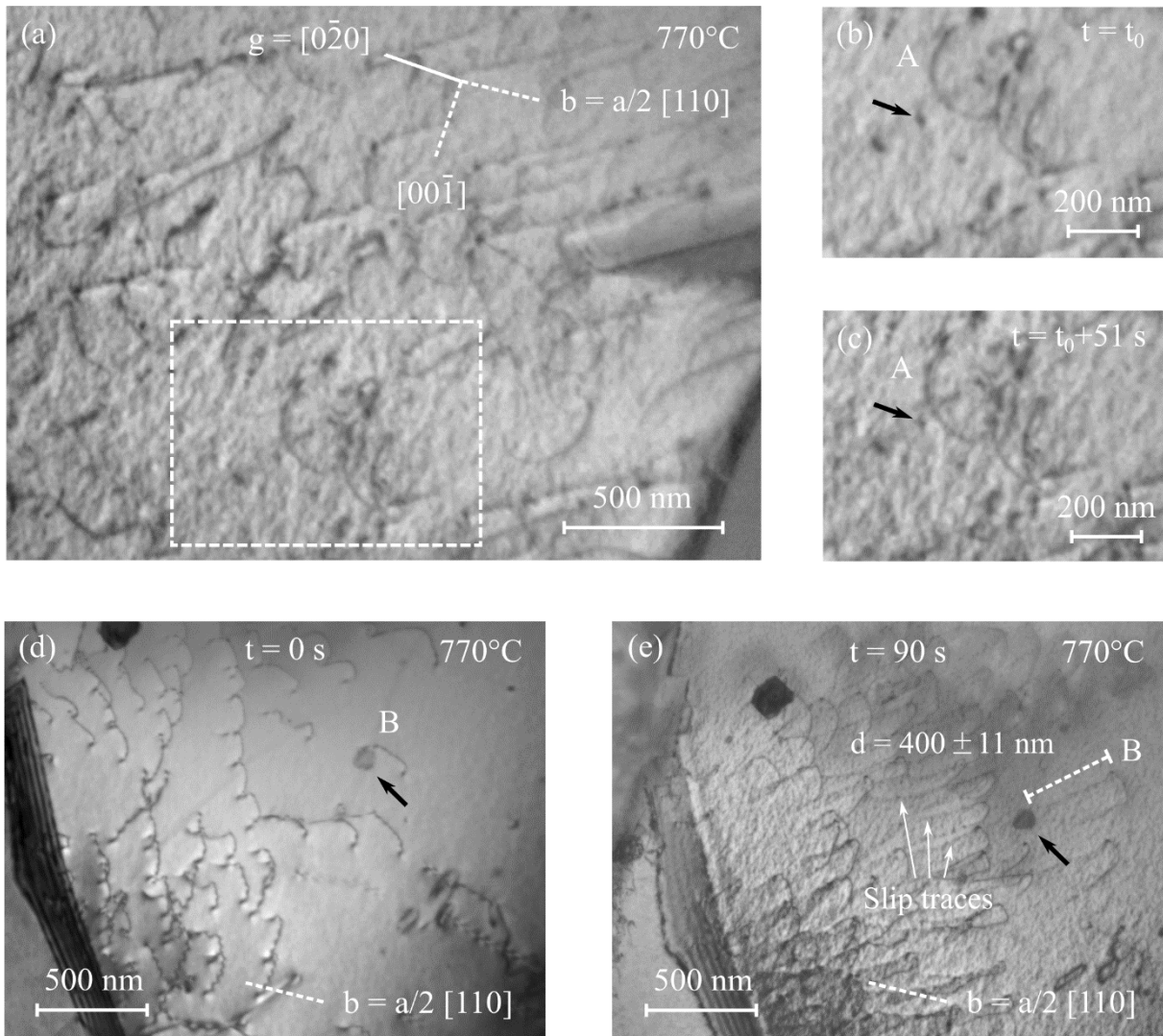


Figure 3. Example of glide mechanism observed during an in-situ TEM experiment at 770°C. (a) View of the right part of an investigated γ -TiAl grain. (b-c) Micrographs extracted from a video sequence at $t = t_0$ and $t = t_0 + 51$ s, corresponding to the zone delineated in (a), showing the positions of the dislocation labelled A with respect to a dot coming from contamination (arrowed), allowing measuring a distance travelled by the dislocation of 59 ± 11 nm, which corresponds thus to a speed of 1.2 ± 0.2 nm/s. The video sequence showed that the motion of the dislocation between these two positions was continuous. (d-e) Micrographs of the left part of the same crystalline grain, showing that the dislocation labelled B has moved over 400 ± 11 nm with respect to an arrowed precipitate during the total duration of the holding at 770°C (90 s), corresponding thus to a velocity of 4.4 ± 0.2 nm/s. Examples of parallel slip traces left by dislocations are indicated.

By measuring the evolution of the projected width between two slip traces in a tilt experiment, the movement plane of the dislocations can be deduced without ambiguity. An example of this method is shown in Fig. 4, where the slip traces of the dislocation labelled B have been aligned parallel to the tilt axis using a tilt rotation sample holder, after the in-situ experiments. These observations evidence a pure glide mechanism. In summary, the

dislocations shown in Figs. 3 and 4 move by pure glide with a continuous movement, indicating thus a viscous glide mechanism.

Finally, Figs. 5a-b show two dislocations labelled A and B, as observed during an in-situ experiment at 790°C, which have moved at 0.5 ± 0.05 nm/s and 1 ± 0.05 nm/s, respectively, that is at an average velocity of 0.75 nm/s, by a continuous movement. The tilt experiment of this sample (Figs. 5d-g) revealed that these dislocations moved by mixed climb. Because the dislocation lines are not parallel or perpendicular to the Burgers vector, the dislocations are of mixed character. Note that some portions of the dislocation loops are elongated approximately along close-packed directions (like [101] and [001], see Fig. 5d). Such behavior was previously observed in Ref. [13], where it is reported that dislocation portions elongate almost parallel to $\langle 101 \rangle$ close-packed directions (the $\langle 101 \rangle$ notation meaning that the first two indices can be permuted).

In summary, this study allowed to identify unambiguously mixed climb and viscous glide within γ -TiAl grains, employing for the first time coupled TEM in-situ and stereographic investigations. Average dislocation velocities v_g of 2.8 nm/s at 770°C for glide, and v_c of 0.75 nm/s at 790°C for mixed climb, have been measured. Consequently, the dislocation velocities for glide and mixed climb are of close orders of magnitude. Of course, the velocity of the dislocations is strongly dependent on the driving stress giving rise to their movement. However, during in-situ experiments, the stress applied on the dislocations τ_a induced by the thermal effects in the TEM thin foils is unknown. The method to measure τ_a using the line tension of curved dislocations [20] could not be employed here, because the curved portions disappeared always rapidly. Nevertheless, the stresses induced by the thermal effects in the TEM thin foils is assumed to yield comparable τ_a values in all the experiments, because at the onset of the movement of the dislocations ($v \approx 0$), τ_a can be approximated to the critical resolved shear stress τ_g [20,21] or to the resolved climb stress τ_c . Furthermore, because τ_g and τ_c determined in the post-mortem experiments are close for glide and mixed climb (see Table 1), the same probably holds for the in-situ experiments. Considering these hypotheses, the in-situ investigations indicate that glide and mixed climb occur with similar kinetics. This result is coherent with the fact that dislocations moving by glide and mixed climb coexist in bulk materials investigated by post-mortem TEM (Figs. 1 and 2), as already observed [13], meaning that the propagation of these two dislocation populations occurs at comparable velocities.

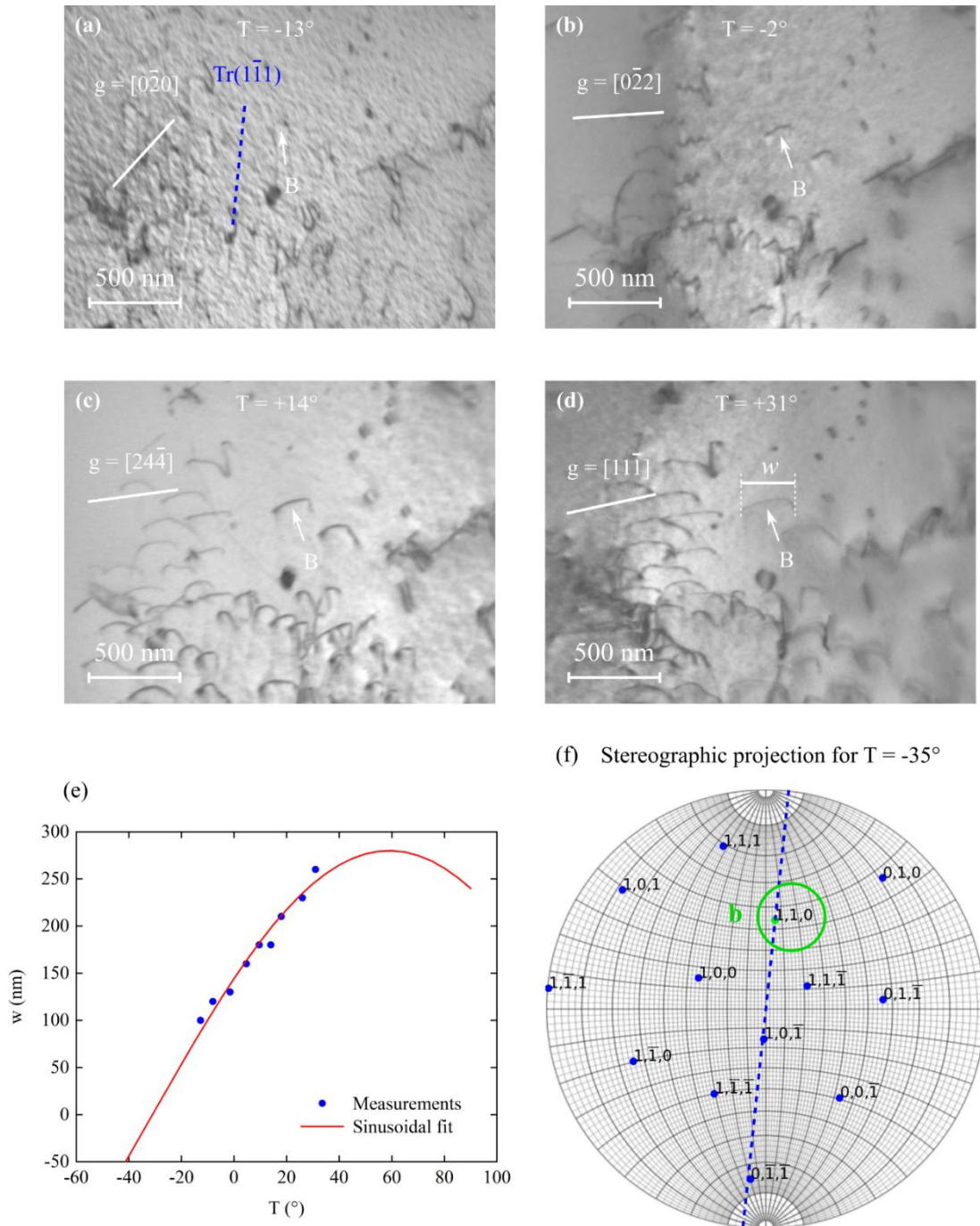


Figure 4. (a-d) TEM micrographs acquired after the in-situ experiment at 770°C, in which the slip traces left on the surfaces of the thin foil by the dislocation labelled B have been oriented parallel to the tilt axis of the sample holder. (e) Evolution of the projected width w of the slip traces as a function of the tilt angle T , allowing to determine the tilt angle for which the movement plane is edge-on ($T = -35^\circ$). (f) Stereographic projection for $T = -35^\circ$, showing that the movement plane is the $(1\bar{1}1)$ plane (blue dotted line, whose trace is presented in view (a)) containing the Burgers vector $\vec{b} = a/2 [110]$ (circled in green), indicating that a glide mechanism is prevailing.

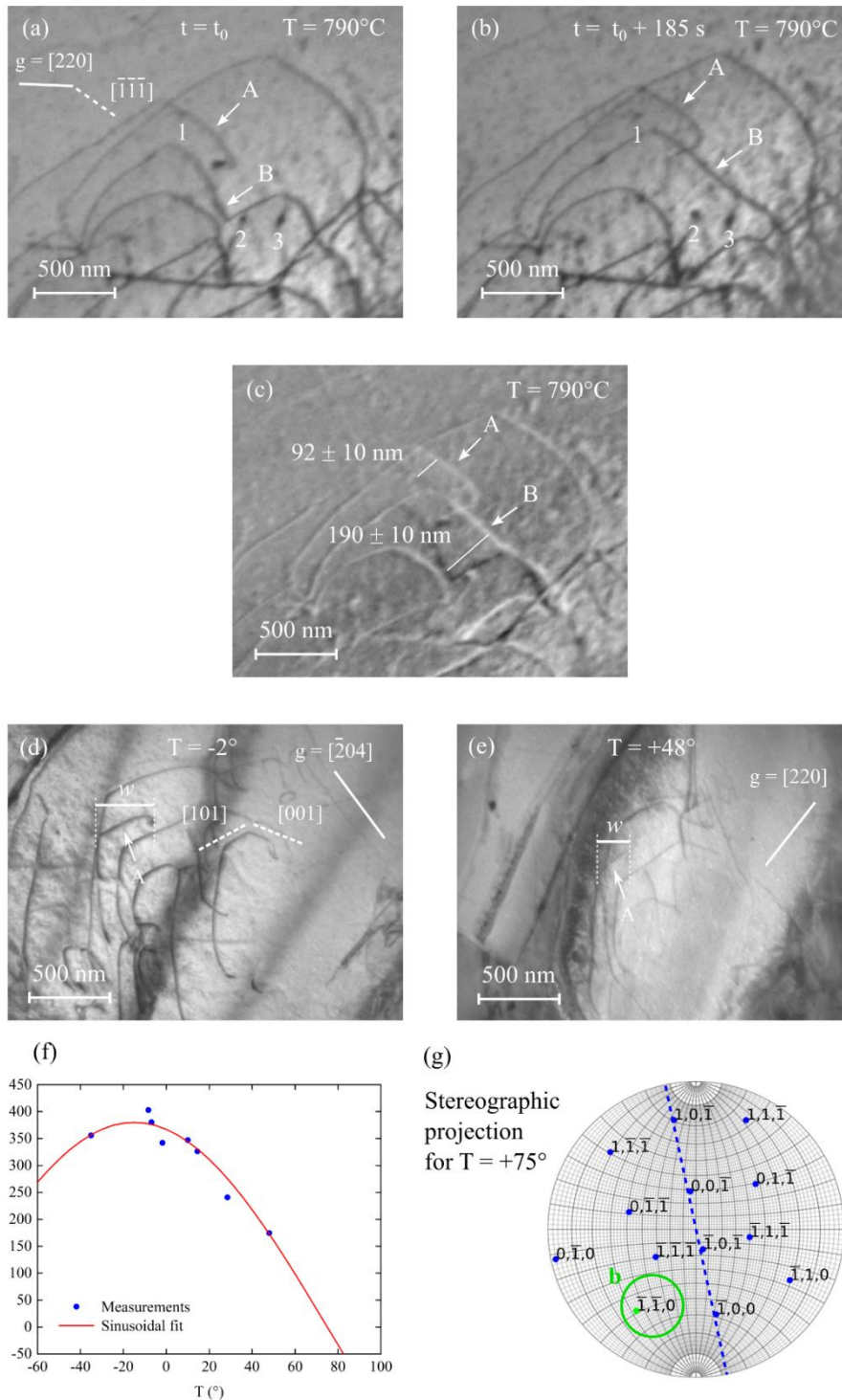


Fig. 5. Example of a climb mechanism observed during a TEM in-situ experiment at 790°C . (a-b) Micrographs extracted from a video sequence at $t = t_0$ and $t = t_0 + 185 \text{ s}$, showing the positions of two dislocations, labelled A and B, with respect to dots coming from contamination labelled 1, 2 and 3. (c) Superposition of micrographs (a) with micrograph (b) in inverted contrast, allowing to see the initial and final positions of the dislocations in black and white, respectively. This shows that the two dislocations A and B have moved over $92 \pm 10 \text{ nm}$ and $190 \pm 10 \text{ nm}$ during 185 s , giving velocities of $0.5 \pm 0.05 \text{ nm/s}$ and $1 \pm 0.05 \text{ nm/s}$, respectively. (d-g) Determination of the movement plane of the dislocation

loop labelled A by measurements (d-e) and plot (f) of the projected width w of the loop at different tilts T , which allows drawing it in the stereographic projection (g). This shows that the movement plane, seen edge-on for $T = +75^\circ$, is close to the $(0\bar{1}0)$ plane (blue dotted line), which does not contain the Burgers vector $\vec{b} = a/2 [\bar{1}\bar{1}0]$, (circled in green) thus, evidencing a mixed climb mechanism.

During the in-situ experiments at 770°C , continuous glide of dislocations of mixed edge-screw character is found (see Figs. 3 and 4). Therefore, this mechanism is different from glide at room temperature of screw segments by jumps between sessile and glissile configurations in $\{111\}$ planes [9]. It is thus more consistent with solute dragging, in which dislocation glide is controlled by diffusion of interstitial solutes segregated in the dislocation cores, as in the case of Zr [20]. When temperature increases, solute dragging occurs usually after dynamic strain ageing, which occurs in TiAl at $400\text{-}600^\circ\text{C}$ [10,11]. Therefore, the observation of glide controlled by solute dragging in TiAl at 770°C is fully consistent with the findings reported in literature. In this binary Ti-48.4Al-0.1B (at.%) alloy, however, no solute atoms were intentionally added (except B, which forms TiB and/or TiB₂ precipitates [22]). However, interstitial contaminants like O, whose content is 573 mass ppm, or also the matrix Ti and Al atoms, exhibit probably enough mobility [23,24] to segregate in the dislocation cores and to follow their displacement, inducing a continuous movement. Concerning the dislocations moving by mixed climb, their elongation approximately along close-packed directions is related to slow nucleation of jog pairs, followed by their lateral motion by climb at higher kinetics [25]. These directions are of $\langle 101 \rangle$ type, as already reported by [13], but it seems that $[001]$ directions are also possible, as well as more general directions.

In conclusion, dislocations moving by glide and mixed climb are found to coexist in a binary near- γ Ti-48.4Al-0.1B (at.%) alloy exposed to creep at 800°C under 36 MPa, as shown by post-mortem TEM investigations. Dislocation velocities were measured by in-situ TEM experiments at $770\text{-}790^\circ\text{C}$, simultaneously with the determination of the involved movement planes, in order to establish the appearing mechanism (glide or mixed climb). Thus, pure glide of screw-oriented dislocation segments was found to occur at an average velocity of v_g of 2.8 nm/s with a continuous movement consistent with a solute dragging mechanism. Mixed climb was also observed to take place at an average velocity v_c of 0.75 nm/s, that is, in the same order of magnitude as in the case of glide. This shows that, even if in the general case, v_c is lower than v_g , as stated in the classical theories [1,2], v_c can reach v_g in a transition temperature domain, leading to dislocation segments moving by climb and mixed climb coexisting in comparable proportions.

Acknowledgments

This work was supported by the cooperative Austrian-French project HITIAL-18-CE91-0008-01 project co-supported by the French Agence Nationale de la Recherche (ANR) and the Austrian Fonds zur Förderung der wissenschaftlichen Forschung (FWF), and partially

financed by the grant NanoX n° ANR-17-EURE-0009 in the framework of the “Programme des Investissements d’Avenir”. Michael Musi is a Recipient of a DOC Fellowship of the Austrian Academy of Sciences at the Department of Materials Science, Montanuniversität Leoben.

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