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ESCUELA TÉCNICA SUPERIOR DE
INGENIEROS DE CAMINOS, CANALES Y PUERTOS

**Micromechanics of fully lamellar TiAl alloys:
effect of lamellar orientation and width**

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effect of lamellar orientation and width**

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A mi madre y mi padre,
a Carla, y a Pablo.

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Abstract

Titanium aluminide alloys with Al concentrations between 35 and 49 at.% (γ -TiAl alloys) exhibit a wide range of microstructures formed by the two intermetallic phases γ (TiAl) and α_2 (Ti₃Al). Among these microstructures the so-called fully lamellar, consisting on the arrangement of γ and α_2 lamellae into colonies, have the potential to replace heavier materials in high temperature structural applications such as automotive and aerospace engine components due to their excellent mechanical response. The increasing demands on modern turbines require microstructural optimisation of γ -TiAl alloys. However, this is a complex task, as the mechanical behaviour of these materials is determined by multiple microstructural parameters such as the colony size, the lamellar size and orientation, the domain size, etc. Isolating the influence of individual microstructural parameters has, to date, not been achieved successfully. The main scope of this thesis is to understand both the effect that lamellar orientation and thickness have on the response of isolated colonies, as well as dislocation-interface interactions in a Ti-45Al-2Nb-2Mn (at.%) + 0.8(vol.%) TiB₂ (Ti4522XD).

With this aim, first, micropillars with lamellae oriented at 0°, 45° and 90° with respect to the loading direction were compressed at room temperature. The results revealed a large plastic anisotropy, that was rationalised, based on slip/twinning trace analysis, according to the relative orientation of the main operative deformation modes with respect to the lamellar interfaces. Loading at 45° resulted in the activation of soft longitudinal modes, where both the slip plane and the slip direction were parallel to the interfaces, and therefore, little interaction of dislocations with lamellar interfaces take place. At 0° loading, deformation was mainly accommodated by harder mixed deformation modes (with an oblique slip plane but a slip direction parallel to the lamellar interfaces), although the lamellar interfaces seemed to be relatively transparent to slip transfer. 90° loading represented the hardest mode and deformation was accommodated by the activation of transverse deformation mechanisms, confined to individual lamellae, together with longitudinal ones that were activated, due to

their softer nature, despite their very small Schmid factors. In addition, a thorough study of pillar size effects revealed that the results were insensitive to pillar size for dimensions above 5 μm .

Second, a fully lamellar microstructure with a nanoscale lamellae width was developed by, first, heat treating the as-received material within the α domain, followed by rapid cooling at cooling rates of $2000\text{ }^{\circ}\text{C s}^{-1}$ in a Gleeble system, and finally low temperature ageing. The strengthening induced by the nanoscale lamellar thickness, investigated as a function of lamellar orientations by micropillar compression, was found to be even higher than what can be achieved by high Nb alloying.

Finally, a microtensile device, which allows to test specimens as small as 5 μm in length, was designed for the present thesis research. A microtensile specimen of the Ti4522XD alloy was tested in tension and the deformation mechanisms were investigated. The results revealed that lamellar interfaces are relatively transparent to dislocation propagation. Order variant interfaces present fully geometrically compatible slip systems that do not require a change in the Burgers vector of the transmitted dislocation. In contrast, in true twin and pseudo-twin interfaces, the geometrical alignment between incoming and outgoing dislocations is not perfect, but plays an important role on determining which slip systems are activated, being the preferred outgoing system that with the maximum geometrical alignment.

The work presented in this thesis served to successfully isolate the effect of lamellar orientation and width in a γ -TiAl alloy, and to quantify their implication on the mechanical behaviour of isolated colonies. The methodology developed in this work can be further extended to study the effect of other microstructural parameters, whose results can be applied for developing mesoscale plasticity models that capture the micromechanics of fully lamellar TiAl microstructures at larger length scales.

Resumen

Las aleaciones de titanio-aluminio con contenido en Al entre el 35 y 49 at.% (denominadas aleaciones γ -TiAl) presentan una amplia variedad de microestructuras compuestas por dos fases intermetálicas: γ (TiAl) y α_2 (Ti₃Al). La microestructura laminar, formada por colonias compuestas de láminas de γ y α_2 , presenta las propiedades más beneficiosas para la fabricación de componentes de motores de combustión interna y de reacción, los cuales están sujetos a exigentes sollicitaciones termomecánicas. Una creciente demanda de estos componentes requiere de la optimización de dichas aleaciones. Sin embargo, esta tarea es difícil dado que la respuesta mecánica de estos materiales viene determinada por un gran número de parámetros microestructurales que actúan simultáneamente, como el tamaño de colonia, ancho y orientación de láminas, tamaño de dominio, etc. Esta tesis tiene como objetivo aislar el efecto que tienen el ancho y orientación de láminas sobre la respuesta mecánica de colonias aisladas, así como la caracterización de la interacción entre dislocaciones y fronteras de lámina en una aleación Ti-45Al-2Nb-2Mn (at.%) + 0.8(vol.%) TiB₂ (Ti4522XD).

Con este objetivo, en primer lugar, se han comprimido micropilares a temperatura ambiente con láminas orientadas a 0°, 45° y 90° con respecto al eje de carga, confirmando la presencia de una elevada anisotropía plástica. La variabilidad en la respuesta mecánica de micropilares con diferentes orientaciones de láminas ha sido examinada a través de las trazas visibles en la superficie externa de los micropilares, las cuales están asociadas al deslizamiento cristalográfico o maclado responsables de la deformación plástica. En las probetas ensayadas a 45° se ha podido observar la activación predominante de sistemas longitudinales, cuyo plano y dirección de deslizamiento están contenidos en las láminas, siendo estos paralelos a las intercaras, dando lugar a una mínima interacción con las fronteras de lámina. En las probetas ensayadas a 0° se ha podido observar que mayoritariamente se activan sistemas de deformación de tipo mixto, cuyo plano de deslizamiento está contenido en las láminas pero cuya dirección de deslizamiento interseca con las intercaras. A pesar de esto, las fronteras de lámina parecen ser relativamente transparentes a la propagación de

dislocaciones de los sistemas mixtos. Los micropilares orientados a 90° han representado la orientación más dura, activándose sistemas transversales, cuyo plano de deslizamiento y dirección intersecan las fronteras de lámina. Además, se ha podido observar la activación de sistemas longitudinales a pesar de presentar un factor de Schmid muy bajo para estas orientaciones. Adicionalmente se ha llevado a cabo un estudio de efectos del tamaño. Este estudio ha revelado que los resultados obtenidos son independientes de la dimensión de los micropilares fabricados por encima de $5\text{ }\mu\text{m}$. Estos resultados pueden servir para alimentar modelos numéricos de plasticidad multiescala que capturen el comportamiento micromecánico de aleaciones $\gamma\text{-TiAl}$.

En segundo lugar, se ha diseñado una estrategia para reducir el espaciado laminar a escala nanométrica a partir de un tratamiento térmico en el dominio de α , seguido de un enfriamiento ultra-rápido a una velocidad de $2000\text{ }^\circ\text{C s}^{-1}$ en un sistema Gleeble, seguido de un proceso de envejecimiento a baja temperatura. El endurecimiento obtenido a partir de la reducción del espaciado laminar ha sido estudiado en función de la orientación de lámina, confirmando una mejora en la respuesta mecánica superior a la obtenida por adición de Nb.

Finalmente, se ha diseñado un dispositivo que ha permitido estudiar el comportamiento mecánico a tracción de una microprobeta de $\gamma\text{-TiAl}$ fabricada por haz focalizado de iones. Los resultados muestran que las intercaras son relativamente transparentes a la propagación de dislocaciones. Las fronteras de tipo order variant presentan compatibilidad geométrica entre todos sus sistemas de deslizamiento, por lo que la dislocación transmitida mantiene el vector de Burgers. En cambio, las fronteras de tipo pseudo-twin y true twin requieren un cambio en el vector de Burgers en la dislocación saliente, siendo el sistema de deslizamiento más probable aquel que presenta la mayor compatibilidad geométrica.

El trabajo derivado de esta tesis ha servido para aislar y cuantificar el efecto del ancho y orientación de láminas en colonias aisladas de una aleación $\gamma\text{-TiAl}$. Además, la metodología desarrollada puede ser extendida al estudio de otras variables microestructurales, siendo de gran utilidad para el desarrollo de modelos predictivos.

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Nomenclature

Roman Symbols

A	Cross sectional plane
b	Burgers vector
D	Micropillar diameter
F	Force
m	Schmid factor

Greek Symbols

β	Slip plane orientation
ϕ	Angle between the direction of the applied force and the normal to the slip plane
Φ_L	Angle between the direction of the applied force and the lamellar interfaces
λ	Angle between the direction of the applied force and the slip direction
λ_{α_2}	Average lamellar thickness of α_2 (Ti ₃ Al)phase
λ_C	Colony size
λ_D	Domain size
λ_γ	Average lamellar thickness of γ (TiAl)phase
λ_L	Lamellar thickness
$\bar{\lambda}_{max}$	Statistical average length of the weakest single arm dislocation source

λ_S	Size of the operating dislocation source
μ	Anisotropic shear modulus
ρ_{tot}	Dislocation density
σ	Stress
σ_f	Fracture stress
σ_y	Yield stress
τ	Shear stress
τ_{CRSS}	Critical resolved shear stress

Acronyms / Abbreviations

AM	Additive Manufacturing
APB	Antiphase Boundary
BC	Band Contrast
BCC	Body Centred Cubic
BF	Bright Field
BSE	Backscattered Electron
CBED	Convergent Beam Electron Diffraction
CCD	Charge-Coupled Device
DF	Dark Field
DF	Diffraction Pattern
EBM	Electron Beam Melting
EBSD	Electron Backscatter Diffraction
EDX	Energy-Dispersive X-Ray Spectroscopy
FCT	Faced Centred Tetragonal

FEG	Field Emission Gun
FIB	Focused Ion Beam
HAADF	High-Angle Annular Dark Field
HCP	Hexagonal Closed Packed
HIP	Hot Isostatic Pressing
HR-SEM	High-Resolution Scanning Electron Microscopy
HREM	High-Resolution Transmission Electron Microscopy
IM	Ingot Metallurgy
IPF	Inverse Pole Figure
LMIS	Liquid-Metal Ion Source
MAD	Mean Angular Deviation
MMC	Metal Matrix Composite
MP	Micropillar
OD	Ordinary Dislocation
OV	Order Variant
PM	Powder Metallurgy
PST	Polysynthetically Twinned
PT	Pseudo-Twin
RT	Room Temperature
SADP	Selected Area Diffraction Pattern
SAED	Selected Area Electron Diffraction
SD	Superdislocation
SEM	Scanning Electron Microscopy

SE	Secondary Electron
SLM	Selective Laser Melting
SPS	Spark Plasma Sintering
STEM	Scanning Transmission Electron Microscopy
TEM	Transmission Electron Microscopy
TT	True Twin
XRD	X-Ray Diffraction

Chapter 1

Introduction and research objectives

The mechanical behaviour of fully lamellar (FL) gamma titanium aluminides has been extensively studied in the literature since the 1920s (Mishurda and Perepezko; Schuster and Palm). Their excellent properties make them potential candidates to replace Ni superalloys in high temperature structural applications, such as automotive and aerospace engine components. TiAl based intermetallics present low density ($3.9 - 4.2 \text{ g cm}^{-3}$, depending on composition and constitution), high melting temperature, good elevated temperature strength, high modulus, high oxidation resistance and favourable creep properties (Appel et al., b; Clemens and Mayer; Dimiduk; Kim, a,b; Voice). Moreover, they can be processed through conventional manufacturing methods such as casting, forging and machining.

The most promising titanium aluminides are those with Al content ranging between 44 to 48 at.% (Appel et al., b; Clemens and Kestler; Huang and Chesnutt, 1994; Kim, a,b; Kim and Dimiduk, a). They are endowed with excellent mechanical properties that derive, to a large extent, from the volume fraction of the γ and α_2 constituent phases and their characteristic lamellar microstructure, which is formed by grains, so-called colonies, of alternating layers of γ and α_2 phases.

The mechanical response of FL γ -TiAl polycrystals is governed by numerous parameters such as the composition of the alloy, the colony size, the lamella size, the volume fraction of the two phases, and the misorientation and character of the interfaces. Isolating the effect of one specific microstructural feature on the deformation and fracture behaviour is, thus, very

challenging (Muñoz-Moreno et al., b, 2012). Furthermore, identifying dislocation activity in such systems is prevented by the lack of reliable experimental techniques that are suitable for this purpose (Cepeda-Jiménez et al.). In this sense, the difficulty in understanding the influence of microstructure on the activation of specific slip systems is paramount (Muñoz-Moreno et al., a). A better knowledge of the fundamentals of crystallographic slip in these materials could be critical for enhancing alloy development. Moreover, the development of models that can capture reasonably well the mechanical behaviour of FL microstructures requires as input information statistically sound experimental data about the influence of phase distribution, crystallographic orientation and lamellar interface nature on slip activation. Such studies are currently scarce (Lebensohn et al.; Zambaldi et al., a).

This thesis aims at overcoming current limitations in the understanding of the deformation of a fully lamellar γ -TiAl alloy, Ti-45Al-2Nb-2Mn (at.%) + 0.8 (vol.%) TiB₂ (Ti4522XD), by establishing a solid methodology that allows to characterise its mechanical response at the microscale level. With this aim, micrometer scale specimens milled out from single colonies at different lamellar orientations (Φ_L) with respect to the loading direction were compressed. The dimensions of the micropillars were determined according to a size effect study conducted with the aim of extracting data representative of the behaviour of single colonies. The deformed samples were further analysed by SEM and TEM to characterise the slip/twinning systems activated at different lamellar orientations (Φ_L). These results revealed a strong plastic anisotropy and two main modes of deformation: a soft mode for lamellar orientations close to $\Phi_L = 45^\circ$ and two hard modes for $\Phi_L = 0^\circ$ and $\Phi_L = 90^\circ$, respectively. Furthermore, the average lamellae width is expected to play a key role in the yield strength. Thus, the Ti4522XD alloy under study was subjected to an annealing, followed by water quenching and a low temperature ageing treatment, giving rise to the precipitation of lamellae with thickness in the nanoscale regime. Subsequently, the mechanical properties of isolated colonies were further characterised by compression of micropillars, confirming a significant strengthening of the alloy response. Finally, a micron-sized diamond grip was designed with the aim of characterising the behaviour of micrometer scale specimens under tensile loads, allowing to identify the mechanisms governing dislocation propagation across interfaces in fully lamellar γ -TiAl alloys.

The work described above has been structured in the following chapters:

- Chapter 2: Literature review highlighting the physical metallurgy, deformation behaviour and mechanical properties of γ -TiAl alloys.
- Chapter 3: Description of the materials used in this thesis, their preparation for testing and methods applied to characterise their microstructure and mechanical properties.
- Chapter 4: Study of the effect of the lamellar orientation on the mechanical response of isolated colonies through the compression of micropillars. Identification of the main deformation mechanisms, and definition of a critical resolved shear stress (τ_{CRSS}) associated to each slip/twinning system.
- Chapter 5: Definition of a methodology to reduce lamellar thickness distribution to the nanometer regime, consisting of an annealing heat treatment followed by water quenching at very high cooling rates. Analysis of the effect of lamellar refinement on the mechanical response of isolated colonies by compression of micropillars with different lamellar orientations, following the methodology described in Chapter 4.
- Chapter 6: Analysis of the slip activity and mechanical response of individual colonies of FL γ -TiAl alloys under tensile stresses. Design and fabrication of a microtensile specimen and a custom-made diamond grip, operated in an *in situ* instrumented nanoindentation system inside a scanning electron microscope.
- Appendix A: Description of a strategy to process EBSD data so that the slip and twinning systems among lamellae within a single colony with the same $(hkl)[uvw]_\gamma$ indices belong to the same morphological set of systems, i.e., longitudinal, mixed or transverse.
- Appendix B: Calibration of a crystal plasticity subroutine that captures the mechanical response of isolated lamellar colonies using the experimental results of the present work as input.

Chapter 2

Literature review

2.1 Physical metallurgy of γ -TiAl based alloys

2.1.1 Introduction

Titanium aluminide alloys have attracted significant attention from the research community and from industry because they present a unique combination of mechanical properties when evaluated on a density-corrected basis. This makes them excellent candidates for replacing the superalloys currently used in high temperature structural applications. Figure 2.1 compares the specific modulus and specific strength of γ -TiAl alloys with those of other high temperature structural materials. The data presented in the graph highlights the advantageous properties of TiAl alloys, which make them prone to find use in a wide range of components in the automotive, aero-engine and power-plant turbine industries. In particular, they present a low density (3.9 to 4.2 g cm⁻³, depending on composition and constitution), high melting temperature, good elevated temperature strength and high modulus, high oxidation resistance and favourable creep properties (Appel et al., b; Clemens and Mayer; Dimiduk; Kim, a,b; Voice). In addition, they can be processed through conventional manufacturing methods such as casting, forging and machining. However, due to their intermetallic nature, complex microstructure and inherent brittleness, γ -TiAl alloys can suffer from limited ductility at room temperature (RT). In order to overcome this limitation and to enhance their creep strength and resistance to oxidation, several γ -TiAl based alloys with specific types of microstructure and composition have been investigated within the framework of extensive research and development programs (Clemens and Mayer; Kim, a,b).

Fig. 2.1 Comparison of the (a) specific moduli and (b) specific strengths of γ -TiAl alloys with those of other structural materials, as a function of temperature Dimiduk. The data has been redrawn based on the original diagrams.

2.1.2 Binary Ti-Al phase diagram

The first Ti-Al phase diagrams covering the whole concentration range date from the 1950s (Bumps et al., 1952; Ogden et al., 1951). Existing discrepancies between different versions of the diagram derive from the high sensitivity of titanium aluminides to impurities like oxygen and from limitations in identifying the superlattices (Jones and Kaufman; Kattner et al.; Mishurda and Perepezko; Ohnuma et al.). The latest work covering the state of the art in this topic has been summarised by Schuster and Palm and Hecht et al.. The binary Ti-Al phase diagram (Figure 2.2) contains several intermetallic phases, which are attractive for lightweight high-temperature materials (McAndrew and Kessler).

Two main phases have emerged to be the most relevant for high-temperature structural applications, namely γ (TiAl) and α_2 (Ti₃Al). Their crystallographic structures are summarised in Table 2.1, and illustrated in Figure 2.3. The ordered phase γ (TiAl) has a tetragonally distorted face centred cubic lattice (L1₀ structure) where the Ti atoms occupy the vertices and the centres of two opposite faces and the Al atoms sit on the faces of the rest of the faces. Alternate (002)_γ planes are occupied by the two different species. An earlier work by Braun et al. (1995), where the structure of γ (TiAl) is thoroughly reviewed, reported that the strong anisotropic character of Ti bonding might be responsible for the $c:a$ ratio being larger than unity in the L1₀ structure. This, indeed, has a significant effect on the behaviour of

Fig. 2.2 Binary Ti-Al phase diagram according to Schuster and Palm.

this intermetallic, which will be discussed in further sections. The α_2 (Ti₃Al) phase possesses a hexagonal D0₁₉ ordered structure, which is formed via an ordering reaction of the high temperature disordered α phase during cooling.

Table 2.1 Crystallographic data of the two constituent phases in γ -TiAl alloys.

Phase	Space group	Strukturbericht designation	Prototype	Lattice parameters	Composition
γ (TiAl)	P4/mmm	L1 ₀	AuCu	$a = 0.3997$ nm	50 at.% Al ^a
				$c = 0.4062$ nm	
				$a = 0.4000$ nm	50 at.% Al ^b
				$c = 0.4075$ nm	
				$a = 0.4016$ nm	50 at.% Al ^c
α_2 (Ti ₃ Al)	P6 ₃ /mmc	D0 ₁₉	Ni ₃ Sn	$c = 0.4068$ nm	
				$a = 0.5765$ nm	25 at.% Al ^d
				$c = 0.4625$ nm	

^aDuwez and Taylor (1952)

^bBraun et al. (1995)

^cPfullmann and Beaven

^dBlackburn and Williams (1967)

Fig. 2.3 Crystal structures of (a) α_2 and (b) γ phases.

2.1.3 Ternary and multicomponent alloy systems

The ductility and strength of γ -TiAl alloys are modulated by their composition. Since room temperature ductility is a major concern in these alloys, to guarantee machinability of components, these engineering alloys based on the γ (TiAl) phase have a composition in Al ranging between 44 to 48 at.% (Appel et al., b; Clemens and Kestler; Huang and Chesnutt, 1994; Kim, a,b; Kim and Dimiduk, a). The maximum elongation to fracture has been observed in alloys with a composition of Ti-48Al (at.%) (Appel et al., b; Huang and Chesnutt, 1994; Kim, a). Despite this, they cannot generally be used in engineering applications due to their limited creep strength and low resistance to oxidation (Appel et al., b; Clemens and Kestler; Huang and Chesnutt, 1994; Kim, a,b; Kim and Dimiduk, a). Thus, the use of additional alloying elements is needed in order to further improve the mechanical properties. These alloying elements are typically Cr, Nb, V, Mn, Ta, Mo, Zr, W, Si, C, Y and B and their effect is summarised in Table 2.2 (Kothari et al.). Depending on their solubility, they can be incorporated into the constituent phases in solid solution, modifying the energies, planar defects or diffusion coefficients; alternatively, they can lead to the formation of various types of carbides, borides and silicides, promoting precipitation hardening, grain refinement during casting, stabilisation of the microstructure against growth, or transient phases that decompose into fine structures. In general, the solubility of alloying elements is limited to about 2 to 3 at.% as indicated by their respective ternary phase diagrams (Hellwig et al.;

Jewett et al.; Kainuma et al.; Palm et al., 1997; Saunders, 1999; Singh and Banerjee, a,b), being Zr, Nb and Ta exceptional in this sense, as they remain soluble at higher contents, in the case of Nb up to 9 at.% at 1200 °C (Kainuma et al.). An extensive research during the last decades on the effect that alloying elements have on the microstructure and mechanical properties of γ -TiAl alloys, has given rise over time to the development of three generations of these materials.

First generation γ -TiAl alloys

At the beginning of the 90s TiAl alloys were based on the Ti-48Al two phase binary alloy, endowed with the highest elongation to failure at room temperature (Kim, a). The addition of alloying elements like Nb, a β -stabiliser (Table 2.2), led to an enhancement of the high-temperature strength and creep resistance and, simultaneously, to an increase in the resistance of oxidation, by lowering the volume fraction of the α_2 (Ti₃Al) phase, with higher O₂ solubility compared to the γ (TiAl) phase, and by promoting Al₂O₃ formation; the addition of Cr and Mn allowed an increased in the elongation-to-failure at room temperature (Appel et al., b; Huang and Hall; Kim, c). Thus, at the earliest stages, the most investigated alloy was Ti-48Al-2Cr-2Nb, patented by Huang and Latham (1989) and commercialised by General Electric.

Table 2.2 Effect of alloying elements on the mechanical properties of γ -TiAl alloys (Kothari et al.).

Element	Effect
Nb	Increases oxidation and creep resistance in small amounts (2%) Increases high temperature strength if added between 5% and 10%
Ta	Increases oxidation and creep resistance and limits hot cracking
W	Improves oxidation and creep resistance
Cr	Increases ductility if added in small amounts (2%) Increases oxidation resistance if added in the range of 8%
Mn	Increases ductility
V	Increases ductility
Mo	Increases strength and creep and oxidation resistance
B	Refines grain size
C	Increases creep and oxidation resistance

Second generation γ -TiAl alloys

During the late 90s there was an important effort in developing a second generation of γ -TiAl alloys with improved creep resistance and room temperature ductility. The composition of these alloys can be summarised as: $\text{Ti}-(45 - 48)\text{Al}-(1 - 3)\text{X}-(2 - 5)\text{Y}-(< 1)\text{Z}$ (at.%) where $\text{X}=\text{Cr, Mn, V}$; $\text{Y}=\text{Nb, Ta, W, Mo}$; $\text{Z}=\text{Si, B, C}$. The addition of higher amounts of β -stabilisers (Y) (Kainuma et al.; Nakamura et al., 1993; Suzuki et al.; Takeyama et al., 1996), such as Nb, was considered in order to further enhance high temperature capabilities. Additionally, incorporating not soluble elements (Z) (Appel et al., b), which lead to the precipitation of silicides, borides and carbides, facilitated microstructure refinement and, in turn, improved elongation-to-failure values. The addition of the latter is commonly carried out by the patented *in situ* XDTM process, which was devised at the Martin Marietta Laboratories (Christodoulou and Brupbacher, 1990; Kumar and Whittenberger; Kumar et al.) for the fabrication of discontinuously reinforced composites with controlled reinforcement shape, size and volume fraction. This method is commercialised by the Howmet Corporation and has been used for the processing of Al and Cu-based metal matrix composites (MMC), titanium, nickel and iron aluminides as well as refractory metal aluminides. TiAl-XDTM alloys exhibit homogeneous microstructures with refined lamellar colonies due to the addition of TiB₂ particles as reinforcements (Wang et al.). Borides are present along the grain boundaries or inside the grains and exhibit particulate, needle or lacey shapes (Larsen et al.). These alloys exhibit superior strength at high temperature and borides are particularly useful to assist fatigue behaviour (Mercer et al.; Voice et al.). The most relevant 2nd generation alloy is the Ti-45Al-2Mn-2Nb-0.8(vol.%)TiB₂, which was exploited by Rolls-Royce.

Third generation of γ -TiAl alloys

Finally, during the last decade, a 3rd generation of TiAl alloys has been developed with the aim of increasing high-temperature capabilities beyond the limits of the previously developed alloys. For that purpose, higher β -stabiliser concentrations were considered in order to promote β -solidifying mechanisms and then obtain more homogeneous and refined microstructures (Appel et al., b; Clemens et al.; Clemens and Mayer). Moreover, the β phase is soft and ductile at high temperatures, although an explanation for that effect is nowadays a matter of study. Therefore, TNB alloys were developed by adding high contents of Nb (5 – 10%) and small additions of B and C (<1%) and TNMTM alloy families were created by adding Nb and Mo in balanced proportions. These 3rd generation alloys benefit

from enhanced creep resistance, although undesired recrystallisation processes may lead to softening under certain processing conditions.

2.1.4 Phase transformations and microstructures

γ -TiAl alloys solidify either through the beta phase or peritectically. Depending on the processing conditions and alloy composition, the solidification paths can vary to a large extent. Thus, a wide range of microstructures can be obtained, as described in later sections. The complexity of the possible phase transformations is further increased in multicomponent alloys, where alloying elements are used for tailoring the microstructures and mechanical properties to some degree (Kim, b; Kim and Dimiduk, a,b; Yamaguchi and Inui).

This section summarises the most relevant solidification and cooling paths commonly followed in the processing of γ -TiAl alloys as well as their effect on the grain structure, texture and microstructure.

2.1.4.1 Solidification

Two-phase alloys based on γ (TiAl) can be obtained either by solidification through the β phase or, for a narrow range of Al concentrations, through peritectic reactions (Figure 2.4).

Fig. 2.4 Section of the binary Ti-Al phase diagram according to the thermodynamic evaluation of Witusiewicz et al..

Al lean γ alloys with Al composition below 45 at.% Al solidify solely through the β phase (body centred cubic structure, BCC, A2 structure and its order B2 variant, β_0) (McCullough et al.). The path followed during solidification and subsequent cooling is $L \rightarrow L+\beta \rightarrow \beta \rightarrow \beta+\alpha \rightarrow \alpha$ where during the $\beta \rightarrow \beta+\alpha$ transformation, crystallographically oriented α

plates obeying the Burgers relationship $\{110\}_\beta \parallel (0001)_\alpha$ and $\langle 111 \rangle_\beta \parallel \langle 11\bar{2}0 \rangle_\alpha$, are precipitated from the β phase. Microsegregation values significantly lower than those predicted by the Gulliver-Scheil equation indicate backdiffusion in the β phase and solidification relatively close to equilibrium (Gulliver, 1922; Scheil, 1942). Cast alloys following a β solidification route exhibit a homogeneous structure governed by relatively large equiaxed grains (Küstner et al.). This grain structure is also observed in ternary and multicomponent β -solidified alloys, with a refinement of the grain size when B is added (Küstner et al.).

In alloys with Al concentrations between 45 and 49 at.% solidification leads to the formation of β dendrites, followed by the peritectic formation of the α phase. For Al concentrations above 49 at.% the hexagonal α phase is the first phase to form and it later transforms into the γ phase through a peritectic reaction (McCullough et al.). Peritectically solidified alloys with high Al contents have been reported to present a characteristic structure based on columnar grains which grow in the opposite direction to that of heat extraction (Küstner et al.). In general, peritectic solidification alloys result in beneficial textured cast components. However, since peritectic γ -TiAl alloys are prone to segregation, their industrial interest remains a matter of controversy (Appel et al., b; Clemens and Mayer).

2.1.4.2 Solid-state transformations

Texture and microstructure of γ alloys is controlled by subsequent solid-state transformations after the solidification process. Depending on composition, the evolution of the microstructure upon cooling will involve passing through the α region, when the Al content is high, or through the β and α regions when Al content is low. Cooling from the α field at very high rates, larger than about 300°C s^{-1} and above, leads to a transformation to the ordered α_2 phase. Lower cooling rates lead to different solid-state transformations, described by Denquin and Naka: (i) lamellar transformation, (ii) discontinuous coarsening and (iii) massive-type transformation. The lamellar transformation leads to microstructures formed by colonies of alternating γ and α_2 lamellae which, when properly refined, are endowed with the most beneficial combination of mechanical properties (Bunshah et al.; Fu and Yoo, c; Kelly et al.; Kim and Dimiduk, a; Lehoczký; Was and Foecke). In the following sections the microstructures resulting from the lamellar transformation and the massive-type transformation, which are the most relevant for the present study, are reviewed.

Lamellar structures in γ -TiAl alloys (cooling rate $< 50^\circ\text{C s}^{-1}$)

Lamellar colonies consist of stacks of platelets of the α_2 and γ phases formed during cooling at slow and medium cooling rates (below 50°C , see Figure 2.5) (Blackburn, 1970; McCullough et al.; Ramanujan; Yamaguchi and Umakoshi). The transformation sequence follows $\alpha \rightarrow \alpha + \gamma \rightarrow \alpha_2 + \gamma$, and the precipitation of lamellae starts to occur in the two phase ($\alpha + \gamma$) region. For Al-lean alloys, the phase formation sequence follows $\alpha \rightarrow \alpha_2 \rightarrow \alpha_2 + \gamma$, resulting in the same lamellar microstructure (Denquin et al., 1993; Jones and Kaufman; Kim, d; Yamabe et al.).

Fig. 2.5 (a) Start temperatures for the formation of lamellar microstructures and the massive transformation in the alloy Ti-47.5Al calculated by Rostamian and Jacot. In addition, experimental results determined by Veeraraghavan et al. (b) are shown. The material had been heat treated at 1400°C , that is, 25°C above the α -transus temperature. (b) Experimental start and finish temperatures for the formation of lamellar microstructures in the alloy Ti-48Al-2Cr-2Nb after a heat treatment at 1380°C (Charpentier et al.).

The transformation from α_2 or α to γ occurs through a change in the lattice structure from hexagonal (HCP) to face centred tetragonal (FCT). This has been observed to take place through the propagation of Shockley dislocations by Xu et al. (1995) and Denquin and Naka. Simultaneously, diffusion leads to an enrichment of the resulting γ -phase with aluminium.

The crystallographic relation between the α_2 and γ phases follows the Blackburn-orientation relationship (Blackburn, 1970) $\{111\}_\gamma \parallel (0001)_{\alpha_2}$ and $\langle 1\bar{1}0 \rangle_\gamma \parallel \langle 11\bar{2}0 \rangle_{\alpha_2}$ ^a, illustrated

^a $\{hkl\}$ and $\langle uvw \rangle$ are often used to differentiate the first two indices from the non-equivalent third one of the tetragonal structure.

Fig. 2.6 Blackburn orientation relationship between α_2 and γ platelets within the lamellar microstructure.

in Figure 2.6. Since there is only one set of basal planes in the hexagonal structure, only one colony of parallel ($\alpha_2 + \gamma$) lamellae can be formed from each prior α grain. According to the Blackburn relationship the most densely packed planes, *i.e.* $\{111\}_\gamma$ planes, are aligned parallel to the basal plane of the γ - or γ_2 -phase, *i.e.* $(0001)_{\alpha_2}$ following $\langle 1\bar{1}0 \rangle_\gamma \parallel \langle 11\bar{2}0 \rangle_{\alpha_2}$. Given the tetragonality of the $L1_0$ structure, three different types of interfaces between adjacent γ lamellae are possible, which can be described by rotations of 60° , 120° and 180° around the normal direction to the $\{111\}_\gamma$ interfacial plane. These interfaces (Table 2.3) are termed pseudo-twin (60°), order variant (120°) and true twin (180°) (Yamaguchi and Umakoshi). Additionally, domain boundaries can be formed during cooling, which divide each γ lamellae in different regions. These boundaries do not exhibit any preferential alignment with any crystallographic habit plane, and have been found to be of order variant (120°) type (Inui et al., 1992b; Yamaguchi et al.; Yang and Wu; Zghal et al.).

In general, lamellar microstructures are polycrystalline (Figure 2.7). Depending on the volume fraction of equiaxed γ grains accompanying the lamellar colonies, lamellar

Table 2.3 Classification of the three distinct γ/γ interface types.

	Inui et al. (1992b)	Generated by	Minimum rotation of
Pseudo-twin	$[\bar{1}\bar{1}0] \nearrow \swarrow [\bar{1}01]$	60° about $[111]_\gamma$	60° about $[\bar{1}\bar{1}\bar{1}]_\gamma$
Order Variant	$[\bar{1}\bar{1}0] \nearrow \nearrow [\bar{1}01]$	120° about $[111]_\gamma$	90° about $[010]_\gamma$
True twin	$[\bar{1}\bar{1}0] \nearrow \swarrow [\bar{1}\bar{1}0]$	180° about $[111]_\gamma$	70° about $[\bar{1}\bar{1}0]_\gamma$

microstructures are termed fully lamellar (absence of equiaxed γ grains), nearly lamellar (small fraction of equiaxed γ grains), duplex (similar fractions of lamellar colonies and equiaxed γ grains) and Widmannstätten. Lamellar microstructures can be additionally processed in the form of polysynthetically-twinned crystals, consisting of a single lamellar colony with densely packed γ lamellae.

Fig. 2.7 Scanning electron micrographs (backscattered electrons imaging) showing (a) the interior of a single colony of a fully lamellar microstructure, (b) a nearly lamellar microstructure, (c) a duplex microstructure and (d) Widmannstätten γ plates.

Massive transformations (cooling rate between $50\text{ }^{\circ}\text{C s}^{-1}$ and $300\text{ }^{\circ}\text{C s}^{-1}$)

Massive transformations were investigated as an alternative route to refine grain size in γ -TiAl alloys, instead of relying in alloying elements like B, as the fragile nature of borides limits the low and intermediate temperature ductility.

A massive transformation consists of short-range diffusion taking place discontinuously behind a migrating reaction front without a change in composition. Several studies have been conducted with the aim of massively transforming α grains to γ using a wide range of cooling rates (Dey et al., 2007, 2009; Hu et al., 2004; Huang et al.; Jiang et al., 2010; Jones and Kaufman; Ramanujan; Sankaran et al., 2009; Takeyama et al., 1993; Veeraraghavan and Vasudevan, 1995; Veeraraghavan et al., a,b; Wang et al., 1992; Yamabe et al.; Zhang et al.). The critical cooling rates that promote massive transformations in TiAl vary with alloy composition (Figure 2.5). The resulting microstructures consist of irregular, mottled γ grains.

Very fast cooling (cooling rate $\gg 300\text{ }^{\circ}\text{C s}^{-1}$)

The use of extremely high cooling rates leads to the retention of the high-temperature α - or α_2 -phase, as reported by Sun. This author showed that ageing of these high temperature metastable phases could lead to the formation of nanosised $\alpha_2 + \gamma$ lamellar microstructures with enhanced mechanical properties.

Exploring the microstructures resulting from ultrafast cooling and annealing, and their associated properties, has gained increased importance with the advent of additive manufacturing (AM) technologies, in which a laser source is utilised to consolidate metallic powders to form 3D components (Herzog et al., 2016). The solidification rates involved in AM technologies range from 10^3 to $10^8\text{ }^{\circ}\text{C s}^{-1}$.

2.1.5 Processing of γ -TiAl based alloys

Titanium aluminides inherent room temperature brittleness makes them challenging and costly to process. γ -TiAl alloys have conventionally been processed by ingot (IM) and powder (PM) metallurgy (Appel et al., b; Gerling et al., 2004; Kothari et al.; Loria, 2001; Wu, 2006). Subsequent post-processing steps are usually required to reach homogeneous microstructures and to avoid remnant porosity. More recently, new high temperature routes based on near net shape techniques are being developed in order to reduce costs by avoiding material losses and final machining. Examples of these processing routes are centrifugal casting, hot isostatic

pressing (HIP) or spark plasma sintering (SPS) (Matsugi et al., 1996; Molénat et al., 2007; Voisin et al., 2013; Yang et al., 2013).

Future advances in processing of γ -TiAl alloys include additive manufacturing (AM) technologies. Additive manufacturing of γ -TiAl alloys is currently mostly being carried out by electron beam melting (EBM), although significant efforts are being invested in optimising selective laser melting (SLM) processes to produce parts with reduced porosity (Biamino et al., 2011; Murr et al., 2010). The Italian company AVIO is currently leading the progress of EBM of γ -TiAl.

2.2 Deformation behaviour of lamellar γ (TiAl) based alloys

2.2.1 Deformation mechanisms

The dominant deformation mechanisms in γ (TiAl) alloys are dislocation slip and twinning. Dislocation slip occurs preferentially in $\{111\}_\gamma$ planes along close-packed $\langle 110 \rangle_\gamma$ directions, which correspond to translation vectors in the $L1_0$ structure. In fully lamellar γ -TiAl alloys, slip systems can be further classified attending to their level of interaction with the interfaces (Lebensohn et al.; Mecking et al.) (Table 2.4). In longitudinal systems the shear plane and direction are parallel to the interfaces; in mixed systems the plane intersects with the interfaces, but their direction remains parallel to these; and in transverse systems, both their shear plane and direction intersect the lamellar interfaces.

Since $\langle 110 \rangle_\gamma$ and $\langle 101 \rangle_\gamma$ directions are crystallographically not equivalent due to the long-range ordering of Ti and Al atoms in the tetragonal structure, two types of dislocations are distinguished: ordinary or unit dislocations (OD), with Burgers vector $\bar{b}_o = 1/2\langle 1\bar{1}0 \rangle_\gamma$, and superdislocations (SD), for which $\bar{b}_s = \langle \bar{1}01 \rangle_\gamma$ and $\bar{b}_s = 1/2\langle 11\bar{2} \rangle_\gamma$ (Appel et al., b) (Figure 2.8). Superdislocations can be further described as two partial dislocations joined by an antiphase boundary stacking fault (APB) in the f.c.t. lattice, *i.e.* $\bar{b}_s = \langle \bar{1}01 \rangle_\gamma = 1/2\langle \bar{1}01 \rangle_\gamma + APB + 1/2\langle \bar{1}01 \rangle_\gamma$ and $\bar{b}_s = 1/2\langle 11\bar{2} \rangle_\gamma = 1/2\langle 10\bar{1} \rangle_\gamma + APB + 1/2\langle 01\bar{1} \rangle_\gamma$.

The activation of these two main types of dislocations, *i.e.* ordinary and superdislocations, is a complex function of temperature and alloy chemistry. For γ -TiAl alloys with near-stoichiometric compositions, deformation at room temperature is mainly accomplished through ordinary dislocations (Inui et al., 1992b). An increase in Al content as well as high levels of interstitial impurities in the γ -phase appear to change this behaviour, making it

Fig. 2.8 (a) Schematic of the γ (TiAl)-phase unit cell, with its slip systems labeled in (b).

easier to activate superdislocation systems (Aindow et al., 1990; Inui et al., 1997; Kad and Fraser, 1994).

The plastic deformation in near-stoichiometric compositions is difficult to assess since α_2 (Ti₃Al) and order domains are formed under these conditions. Thus, no single crystals can be grown for Al-lean γ -TiAl alloys, limiting the understanding of their deformation mechanisms. Micropillar compression tests, which may be conducted within single colonies, offer an alternative to facilitate the identification of deformation systems (see following Chapters).

Twinning in γ (TiAl) alloys takes place along four $\{111\}_\gamma \langle 11\bar{2} \rangle_\gamma$ systems (Table 2.5). This mechanism is considered important to enhance ductility and seems to be promoted by the addition of niobium, which reduces the stacking fault energy in γ (TiAl) (Appel et al., b). Twinning has been found to be virtually insensitive to temperature up to 800 °C (Kishida et al., 1998) and is considered to be assisted by the stress concentrations close to the interfaces in polycrystalline specimens (Appel, 2005; Appel et al., a; Hsiung et al., 2002). Longitudinal twinning has been confirmed to prevail in micropillars tested at room temperature in soft orientations (Edwards et al.).

Table 2.4 Classification of slip systems in γ (TiAl) (Lebensohn et al.; Mecking et al.).

System	Morphology-based	Mechanism-based
(111)[$\bar{1}\bar{1}0$]	longitudinal	ordinary slip
(111)[$0\bar{1}1$]	longitudinal	super slip
(111)[$\bar{1}01$]	longitudinal	super slip
($\bar{1}\bar{1}1$)[$\bar{1}\bar{1}0$]	mixed	ordinary slip
($\bar{1}\bar{1}1$)[$0\bar{1}1$]	mixed	super slip
($\bar{1}\bar{1}1$)[$\bar{1}01$]	mixed	super slip
($\bar{1}\bar{1}1$)[110]	transversal	ordinary slip
($\bar{1}\bar{1}1$)[$\bar{1}10$]	transversal	ordinary slip
($\bar{1}\bar{1}1$)[011]	transversal	super slip
($\bar{1}\bar{1}1$)[101]	transversal	super slip
($\bar{1}\bar{1}1$)[011]	transversal	super slip
($\bar{1}\bar{1}1$)[101]	transversal	super slip

Table 2.5 Classification of twinning systems in γ (TiAl) (Lebensohn et al.; Mecking et al.).

System	Morphology-based	Mechanism-based
(111)[11 $\bar{2}$]	longitudinal	twinning
($\bar{1}\bar{1}1$)[11 $\bar{2}$]	transversal	twinning
($\bar{1}\bar{1}1$)[$\bar{1}1\bar{2}$]	transversal	twinning
($\bar{1}\bar{1}1$)[$\bar{1}\bar{1}\bar{2}$]	transversal	twinning

The propensity of slip (or twinning) in a particular plane is best expressed in terms of the shear stress (τ) required to overcome a characteristic critical resolved shear stress (τ_{CRSS}). The shear stress can be expressed as Equation 2.1:

$$\tau = \sigma \cdot \cos \phi \cdot \cos \lambda \quad (2.1)$$

where $\sigma = F/A$ is the applied normal stress acting on the cross-sectional plane A of the sample, ϕ is the angle between the direction of the applied force F and the normal to the slip plane, and λ represents the angle between F and the slip direction.

The τ_{CRSS} values corresponding to single crystals of γ (TiAl) present a strong variation with Al at.% content and with temperature (Appel et al., b; Fujiwara et al., 1990; Inui et al., 1997). In particular, an increase in the Al content results in an increase of the τ_{CRSS} values. Contrary to conventional disordered materials, where the τ_{CRSS} decreases as the temperature rises, the τ_{CRSS} values of both ordinary τ_{CRSSo} and superdislocations τ_{CRSSs} present an anomalous behaviour with temperature, with a peak between 700 °C and 1000 °C

(Appel et al., b; Fujiwara et al., 1990; Inui et al., 1995). These findings are illustrated in Figure 2.9.

Fig. 2.9 Variation of τ_{CRSS} with temperature for orientations $[001]_\gamma$ (a), $[010]_\gamma$ (b), $[\bar{1}10]_\gamma$ (c) and near $[021]_\gamma$ (d). $\circ$ Ti-56Al (at.%) (Kawabata et al., 1985, 1990), ∇ Ti-56Al (Inui et al., 1997), $\triangle$ Ti-55.5Al (Zupan and Hemker, 2003), $+$ Ti-56Al (Stucke et al.), $\square$ Ti-56Al (Feng and Whang), $*$ Ti-54.5Al (Jiao et al., 2001).

In the $\alpha_2(\text{Ti}_3\text{Al})$ phase, dislocation glide takes place on prismatic, basal and pyramidal slip systems (Figure 2.10) which have increasing critical resolved shear strengths. In lamellar microstructures, basal slip systems constitute longitudinal deformation modes, while prismatic and pyramidal are considered transversal (Table 2.6). The room temperature τ_{CRSS} values of the slip systems operative in $\alpha_2(\text{Ti}_3\text{Al})$ are given in Table 2.7 (Inui et al., 1995; Kishida et al.).

The τ_{CRSS} values corresponding to lamellar microstructures are, to date, not well known. It is very likely that the presence of a large number of complex interfaces leads to very different values than those reported for their monolithic constituents. In order to build powerful models that could help to optimise the behaviour of γ -TiAl alloys, it is deemed essential that τ_{CRSS} values of slip and twinning systems in lamellar microstructures are investigated.

Fig. 2.10 Illustration of the basal $(0001)_{\alpha_2}$, prismatic $(\bar{1}010)_{\alpha_2}$ and pyramidal $(2\bar{1}\bar{1}1)_{\alpha_2}$ deformation systems of $\alpha_2(\text{Ti}_3\text{Al})$.

Table 2.6 Classification of slip systems in $\alpha_2(\text{Ti}_3\text{Al})$.

System	Type
$(0001)[\bar{2}110]$	basal
$(0001)[1\bar{2}10]$	basal
$(0001)[11\bar{2}0]$	basal
$(1\bar{1}00)[11\bar{2}0]$	prismatic
$(01\bar{1}0)[\bar{2}110]$	prismatic
$(10\bar{1}0)[1\bar{2}10]$	prismatic
$(11\bar{2}1)[\bar{1}\bar{1}\bar{2}6]$	pyramidal
$(1\bar{2}11)[\bar{1}\bar{2}\bar{1}6]$	pyramidal
$(2111)[2\bar{1}\bar{1}6]$	pyramidal
$(\bar{1}\bar{1}\bar{2}1)[11\bar{2}6]$	pyramidal
$(\bar{1}\bar{2}\bar{1}1)[1\bar{2}16]$	pyramidal
$(2\bar{1}\bar{1}1)[\bar{2}116]$	pyramidal

Table 2.7 τ_{CRSS} values of the slip systems operative in $\alpha_2(\text{Ti}_3\text{Al})$.

Alloy composition	Pyramidal	Basal	Prismatic	Reference
Ti-36.5Al, 20 °C	900 MPa	320 MPa	100 MPa	Kishida et al. Inui et al. (1995)

2.2.2 Interface energies

It is well documented in the literature that the extraordinary mechanical properties associated to fully lamellar γ -TiAl alloys derive, to a large extent, from the structure and chemical environment of their interfaces, which determines how plastic strain is accommodated (Bunshah et al.; Fu and Yoo, c; Kelly et al.; Lehoczký; Was and Foecke). It is widely accepted that the vast majority of plastic deformation in lamellar TiAl structures is facilitated by γ/γ interfaces, while α_2/γ interfaces are thought to be less favourable for dislocation transfer, since pyramidal slip requires extremely high shear stresses (Appel and Wagner; Appel et al., a; Singh et al., a,b; Sriram et al.; Vasudevan et al.; Wiezorek et al.).

Given the tetragonality of the L1_0 structure and its atomic arrangement, γ/γ interfaces present different interfacial energies (Γ). Previous results obtained through simulations estimate a ratio between the interfacial energies corresponding to the three γ/γ variants ($\Gamma(180^\circ):\Gamma(120^\circ):\Gamma(60^\circ)$) ranging from 1:2:3 to 1:6:7 (Fu and Yoo, a,b,c; Fu et al.; Girshick and Vitek; Hug et al., 1989; Inui et al., 1992a; Lu et al.; Panova and Farkas; Rao et al., 1994; Simmons et al., a,b; Stucke et al.; Wiezorek and Humphreys; Woodward et al.; Yoo and Fu, a,b). These findings agree with the fact that true twin variants, with the lowest associated interfacial energy, are fully coherent boundaries, across which only a change in the stacking sequence of $(111)_\gamma$ planes takes place. The interfacial energies of order variants and pseudo-twin interfaces are raised due to incompatibilities in the c and a parameters, which lead to the formation of misfit dislocations and ledges between regions of good atomic registry. Only in very thin lamellae a long-range atomic order is kept, being the misfit strains taken up by extremely high elastic stresses, homogeneously distributed along the lamellae (Shoykhet et al.).

2.3 Mechanical properties of γ -TiAl based alloys

The $\alpha_2(\text{Ti}_3\text{Al})$ phase is known to be brittle, which is often associated to the slip geometry of its hexagonal ordered D0_{19} structure. However, the $\gamma(\text{TiAl})$ phase is softer due to a larger

number of slip systems available in the tetragonal $L1_0$ structure. The mechanical behaviour of γ -TiAl alloys is highly dependent on their microstructures and the volume fraction of their constituent phases. In general, fully lamellar alloys present high strength and excellent creep resistance, whereas duplex structures are softer and are endowed with higher room temperature elongation-to-failure. The properties of lamellar γ -TiAl alloys are, furthermore, highly dependent on their complex multi-layered configurations, defined by γ/γ and α_2/γ interfaces and, additionally, by colony boundaries in polycrystalline materials. Depending on the interface type and lamellar thickness distributions, as well as on the lamellae orientation with respect to the loading axis (Φ_L), variations in the mechanical behaviour are to be expected.

Lamellar microstructures such as polysynthetically twinned (PST) crystals exhibit pronounced anisotropy of their yield stress and elongation-to-failure. Their magnitudes are highly dependent on the lamellar orientation with respect to the loading axis (Φ_L), as indicated in Figure 2.11. Previous studies conducted on PST crystals (Fujiwara et al., 1990; Hazzledine and Kad; Inui et al., b, 1992b, 1995; Kim et al., 1999a; Nomura et al., 1999; Umakoshi and Nakano; Umakoshi et al.; Yao et al., 1995) with different orientations indicate that specimens with lamellae parallel or perpendicular to the loading axis exhibit higher yield stresses and lower elongation values. In these hard loading modes shear is mostly transverse to lamellae and dislocation pile-ups occur at the interfaces. When Φ_L spans from 30° and 60° , lower yield stress values and higher elongations are registered. In this softer mode, shear occurs parallel to the lamellae interfaces and pile-ups form preferentially at the domain boundaries; dislocation slip paths are larger and a higher ductility is achieved. Figure 2.11 illustrates the stress-strain curves and deformation mechanisms associated hard and soft loading modes. Finally, Yamaguchi (1991) studied the deformation behaviour of a nearly stoichiometric TiAl PST in tension and compression, reporting no yield stress tension-compression asymmetry. The absence of asymmetry was attributed to the fact that lamellae which deform by twinning in compression deform by slip in tension and *vice versa*.

The mechanical response of polycrystalline specimens, which are composed by different fractions of soft and hard lamellar colonies, is a complex function of the mechanical behaviour of individual colonies. These materials suffer from large strain incompatibilities at the colony boundaries, a phenomenon that leads to pre-yielding, which seems to be responsible for the lower ductility associated to fully lamellar specimens compared to microstructures containing equiaxed grains. Having a methodology that would allow to probe the mechanical response

of single colonies within a polycrystalline specimen would be an invaluable asset to better understand and predict the mechanical behaviour of polycrystalline γ -TiAl alloys.

Fig. 2.11 Stress-strain curves illustrating the mechanical response of PST γ -TiAl lamellar structure as a function of the relative orientation of the loading axis with respect to the lamellae interface (Inui et al., 1992b). Yield (σ_y) and fracture (σ_f) stress were calculated for these curves by Hazzledine and Kad.

Chapter 3

Materials and methods

This work focuses on the characterisation of the mechanical response of single colonies of the Ti4522XD alloy under study as a function of lamellar thickness and orientation, and under different loading modes (tension versus compression). With this aim, micropillar compression and micro-tensile tests were carried out within isolated colonies of a centrifugally cast γ -TiAl alloy, using a dual-beam focused ion beam (FIB) to fabricate the micrometer scale specimens, that were subsequently tested by means of instrumented nanoindentation systems. Even though the experimental details regarding the fabrication of the micrometer scale specimens, the testing conditions, and the analysis of the results are thoroughly reviewed in each chapter, the complexity and sophistication of the scientific instruments used to accomplish the work require some additional description. This chapter thus introduces the base material that has been the objective of this study and provides a general overview of the fundamentals of the scientific instruments and the characterisation techniques used in this thesis.

3.1 Fully lamellar Ti4522XD

The nominal composition of the alloy under study was Ti-45Al-2Nb-2Mn (at.%) + 0.8(vol.%) TiB₂. This alloy is also known as Ti4522XD (Christodoulou and Brupbacher, 1990; Kumar and Whittenberger; Kumar et al.). The investigated material was processed by centrifugal casting in the form of rectangular cast specimens at ACCESS e.V. TechCenter (Aachen, Germany) (Aguilar et al.) in a Linn Supercast equipment with mould rotation velocity of 250 rpm, mould preheating temperature of 1200 °C, superheat of 20 to 100 °C and ceramic moulds coated with Y₂O₃. The cast specimens were subsequently hot isostatically pressed at

1185 °C and 170 MPa for 4 h to remove any residual porosity. The alloy possessed a lamellar microstructure, with alternating γ and α_2 layers, as depicted in Figure 3.1.

Fig. 3.1 BSE-SEM image of the characteristic fully lamellar microstructure in γ -TiAl alloys, depicting the arrangement of lamellar colonies.

The reported fraction of α_2 and γ was, respectively, 31 and 69 %, with an average lamellar thickness of $\lambda_{\alpha_2} = 319 \pm 39$ nm and $\lambda_{\gamma} = 287 \pm 133$ nm. These lamellae are arranged in a plate-like manner, defining grains, so-called colonies, which aggregate to constitute the bulk material, depicted in Figure 3.1. The measured colony size was 194 ± 121 μm , with values ranging from 52 to 636 μm (Muñoz-Moreno et al., b,c).

3.2 Metallographic sample preparation

Samples with dimensions of approximately $10 \times 10 \times 2$ mm^3 were machined out of the as-received Ti4522XD plates with a diamond disk in a Struers Accutom-5 precision cutting machine. Subsequently, metallographic preparation of the specimens' surfaces consisted of an initial grinding stage using SiC paper with increasingly finer grit sizes, down to 2000 grit, to ensure that the opposite surfaces of interest (those with dimensions of 10×10 mm^2) were parallel; this was followed by consecutive polishing steps with diamond suspensions starting with a particle size of 9 μm until a surface roughness of 1 μm was achieved; finally a chemical-mechanical polishing step was carried out with a solution of colloidal silica (OP-S) and hydrogen peroxide (30 vol.%).

Alternatively, if the surface of the samples was to be mapped by electron backscattered diffraction (EBSD), grinding was carried out using finer grit sizes, down to 4000 grit, and this was followed by a final electropolishing stage in a Struers LectroPol-5 device, using a commercial A3 electrolyte, at room temperature (20 °C), with a flow rate of 10 and a voltage of 40 V during 30 s. Electropolishing is needed since EBSD requires the outer layer of the specimen to be deformation free.

After polishing, the specimens were glued to aluminium pins where they remained fixed during the subsequent micropillar (MP) fabrication and compression stages in order to reduce potential misalignments between the sample and the flat punch that could lead to the underestimation of the measured strengths.

3.3 Microstructure characterisation

The characterisation of the microstructure and initial inspection of the deformed specimens was carried out by several complementary techniques, which are described in the following. This section will firstly introduce the basis of a SEM and its capabilities, focusing on the configurations used for imaging in the present thesis. Furthermore, a description of the EBSD technique is given, with special emphasis on the particular characteristics of the γ -TiAl phase that makes it challenging to obtain high quality orientation maps of the layered structure in fully lamellar titanium aluminides. Secondly, the basis of the FIB working mode is described in general terms, with special attention to its application in the fabrication of TEM foils using the lift-out technique that allows to precisely select the area of interest for inspection. Finally, the TEM imaging modes utilised in this thesis are described.

3.3.1 Scanning electron microscopy (SEM)

The scanning electron microscope (SEM) allows to image the microstructure of a specimen by scanning its surface with a focused beam of high-energy electrons. The electron beam is produced at the top of the microscope by an electron gun and focused toward the sample surface through electromagnetic lenses. When the electrons hit the sample a variety of signals are produced that reveal information about the interaction volume such as sample morphology, chemical composition, and crystalline structure and orientation. The types of signals produced by a SEM include: secondary electrons (SE) (surface topography), and backscattered electrons (BSE) (composition), diffracted backscattered electrons (used to determine crystal structures and orientations), characteristic X-rays (elemental analysis),

visible light (cathodoluminescence), and heat. To obtain an image, the electron beam scans over a selected area of the sample, and the beam position is combined with the detected signal to generate a 2-dimensional image. Figure 3.2 depicts the signals emitted from different depths of the interaction volume.

In this work, SEM imaging was carried out using a FEI Helios NanoLab 600i field emission gun (FEG) dual-beam microscope.

Fig. 3.2 Signals emitted from different depths of the interaction volume in a SEM.

3.3.2 Electron backscatter diffraction (EBSD)

EBSD was extensively used in this thesis. It provides exhaustive information about the texture and grain boundary nature of polycrystalline samples. This technique has evolved fast over the years and currently large surface areas can be mapped in a relatively short time span. Figure 3.3 is a schematic illustrating the fundamentals of this method. A sample with a flat and highly polished surface is placed at an angle of 20° to the incident electron beam. In this configuration, the atoms of the material elastically scatter a fraction of the electrons, with a small loss of energy. Some of these electrons are incident on atomic planes at angles that satisfy the Bragg equation:

$$n \cdot \lambda^{e^-} = 2 \cdot d_{hkl} \cdot \sin \theta \quad (3.1)$$

where n is an integer, λ^{e^-} is the wavelength of the electrons, d_{hkl} is the spacing of the diffracting plane, and θ the angle of incidence of the electrons on the diffracting plane. These diffracted electrons form a set of paired large-angle cones for each diffracting plane. The intersection of these cones with the flat phosphor screen of the detector forms regions of enhanced intensity, so-called bands, that constitute Kikuchi patterns (or electron backscatter diffraction patterns, EBSDs), depicted in Figure 3.4. These patterns are a gnomonic projection of the diffracted cones, making the band edges appear hyperbolic. The centre lines of the Kikuchi bands correspond to where the diffracted planes intersect with the phosphor screen. Each Kikuchi band can thus be assigned to a diffracting plane, and their intersections correspond to zone axes. A specialised computer software analyses each Kikuchi pattern by, first, detecting a number of bands using an optimised Hough transform, and, second, finding the orientation of the phase under investigation, given as an input, that provides the best fit to the detected bands. When the electron beam is scanned following a predetermined path along an area of a polycrystalline sample and the crystal orientation is measured at each point, the resulting map reveals the grain morphology, orientation and boundaries.

Fig. 3.3 Schematic showing EBSD geometry.

In this work an Oxford Instruments NordlysNano electron backscatter detector (EBSD) equipped with AZtec sytem were utilised to extract information about the crystallographic orientation of the colonies under investigation.

Fig. 3.4 Kikuchi patterns corresponding to a γ variant (a) and to an α_2 lamella (b) in a Ti4522XD alloy.

3.3.3 Transmission electron microscopy (TEM)

TEM is a technique that allows for high resolution microstructural imaging. In particular, TEM is suitable for the study of defects such as dislocations, interfaces, grain boundaries, etc. The limit in resolution depends critically on four parameters: the resolving power of the microscope (usually smaller than 0.3 nm), the energy spread of the electron beam (often several eV), the thickness of the specimen (generally below 100 nm), and the composition and stability of the specimen (Goodhew, 2011).

TEM uses high-energy electrons (100 to 400 keV), transmitted through a specimen, to form an image in a fluorescent screen, a photographic film, or a charge-coupled device (CCD). The transmitted beam splits into two signals: inelastically scattered electrons, which are parallel to the incident beam, or diffracted electrons, which undergo elastic scattering and thus change their direction. Different operation modes exist in a TEM that attempt to modify the outgoing electron waves in a way that provides information about the sample such as that extracted from diffraction and phase contrast images. Diffraction contrast, which reveals defect structures, emerges when the electron beam undergoes Bragg scattering, in the form of bright field (BF) or dark field (DF) images depending on whether the selected reflections include the unscattered beam or not (see Figure 3.5). Phase contrast, also known as high-resolution transmission electron microscopy (HREM) can be utilised for studying the crystal structure at atomic resolution. Moreover, diffraction patterns (DP) provide useful information about the space group symmetries in the crystal and about the orientation with respect to the beam path. When the incident beam is converged, Kikuchi patterns

Fig. 3.5 Bright field (BF) contrast image (a) with a selected reflection of $1\bar{1}00_{\alpha_2}$ type and dark field (DF) contrast image in (b).

arise. In convergent beam electron diffraction (CBED) the image formed is useful for sample orientation. This is achieved by tilting the stage of the TEM, following the Kikuchi lines, towards the desired zone axis.

In this thesis a FEG scanning transmission electron microscope (STEM) FEI TalosTM F200, at IMDEA Materials Institute, and a JEOL JEMTM 2100, at the National Laboratory for Electron Microscopy (CNME) of the Complutense University of Madrid, were used.

3.3.4 Correct identification of the orientation of gamma variants

Gamma variants coexisting within a colony in fully lamellar γ -TiAl exhibit very similar diffraction patterns given their pseudo-cubic $L1_0$ structure with a $c:a$ ratio close to unity (see Figure 3.6). Thus, precisely identifying the correct position of their c -axis is complex. Additionally, given that order variant (OV) lamellar interfaces present a misorientation described by a rotation of 90° about $\langle 100 \rangle_\gamma$ it is crucial to suppress potential misindexing issues, since they constitute an artifact in the correct identification of the γ (TiAl) phase crystallographic orientation. Two approaches can help to precisely discriminate between the correct orientation and the pseudo-symmetrical solutions: (i) the analysis of the atomic stacking order by the detection of superlattice reflections or (ii) the detection of the slight tetragonal distortion of the lattice cell (Zambaldi et al., b).

Fig. 3.6 Kikuchi patterns associated with γ variants coexisting within a colony with their associated stereographic projection illustrated below, sharing their $(111)_{\gamma}$ pole. The misorientations between a:b, b:c, a:c are TT, OV and PT, respectively. Note the high similarities between the b:c (OV) EBSPs.

Superlattice reflections are those reflections present in a diffraction pattern that are kinematically extinct in a disordered (or monoatomic) base lattice but arise when this lattice adopts an ordered structure, as the case for the $\gamma(\text{TiAl})$ phase. In general, for an f.c.c. crystal consisting only of a single atomic species, the condition for systematic absence of diffraction intensity is that h, k, l are not all odd or not all even. However, in the tetragonal $L1_0$ structure associated with $\gamma(\text{TiAl})$, the (001) and (110) reflections appear as superlattice reflections given the ordering of Ti and Al atoms in the lattice. In this case, under the condition that the sum of h and k is even and h, k, l are not all even or not all odd, superlattice diffraction can be observed (Dey et al.).

TEM has been used to characterise $\gamma(\text{TiAl})$ variants' orientations through the identification of superlattice diffraction reflections. These reflections can be easily recognised in selected area diffraction patterns (SADP) and allow to determine the c -axis of a given area in a microstructure (Inui et al., 1992b). However, the main limitation of this technique is the small observable sample volume and the complicated and destructive sample preparation.

EBSD patterns present a solid angle about 70 to 100° that hinders the detection of low angle diffraction bands related to superlattice reflections. Because of this, in order to distinguish between pseudo-symmetrical orientations, the automated indexing algorithm should rely on detecting the characteristic tetragonal distortion of the EBSPs. The present

PhD candidate worked closely with Oxford Instruments PLC in order to improve the accuracy of band detection in AZtec software. As a fruit of this collaboration, a new approach to determine the identity of a detected Kikuchi band in terms of phase and reflector family was defined and was implemented as a commercial solution. The devised method consisted on three stages: (i) detection of the the bands through a low resolution Hough transform; (ii) indexing of the detected bands; and (iii) secondary band refinement, taking into account the expected hyperbolic shape of the band. This approach returns the orientation with the lowest fit residual (measured in terms of the mean angular deviation (MAD) that describes

Fig. 3.7 Typical $\gamma(\text{TiAl})$ pattern with 3 pseudo-symmetry related solutions overlaid. The spread in MAD values is typical for this material and this pattern quality. (a) Pattern recorded by the CCD camera; (b) Pattern with correct solution and a MAD of 0.07° ; (c) pattern with incorrect solution and a MAD of 0.52° and (d) pattern with incorrect solution and a MAD of 0.57° . The arrows point at the simulated bands that do not match the experimental pattern in (c) and (d).

Fig. 3.8 The left side of the picture corresponds to the traditional approach; based on a standard Hough transform and indexing algorithm. The right side of the picture corresponds to the Refined Accuracy approach described in the text. (a) and (b) Band contrast and α_2 in magenta. (c) and (d) IPF-Z of the γ phase and, in addition, the α_2 phase in magenta. (e) and (f) depict band contrast with grain boundaries and the three variants overlaid with α_2 phase. The tables in (e) and (f) show the quantification of the types of variants present in the maps, where order variant are the majority in the left due to the contribution of pseudo-symmetric mis-indexing.

the average angular difference between the position of Kikuchi bands in both the simulated and the acquired pattern, see Figures 3.7 with black arrow indicating mistakenly indexed bands in (c) and (d), and 3.8) (Sitzman et al.). For the automatic indexing algorithm to correctly discriminate between the correct and pseudo-symmetrical solutions, the quality of the patterns recorded through the EBSD system needs to be optimum. This was achieved by electropolishing the surface of the specimen, using the conditions described in Section 3.2 of the present chapter, or by milling the area of interest with a low energy beam when mapping smaller areas such as MP sides.

3.4 Micromachining using a focused ion beam (FIB)

Dual-beam FIB-SEM systems are SEMs endowed with FIB capabilities (Figure 3.9) which include ion imaging and a precision machining tool. The ion column is tilted 52° with respect to the electron beam. FIB imaging is carried out by scanning the sample surface with a focused beam of ions. Commercially, the most commonly used beam sources are Liquid-Metal Ion Sources (LMIS) from which the ions (generally Ga^+) are emitted. The Ga^+ ion beam is then accelerated using a potential energy difference of 1 to 50 keV, and focused into the sample surface by electrostatic lenses. When the beam hits the sample, a small amount of material is sputtered as either neutral atoms or sputtered ions, and secondary electrons are emitted. As the beam scans the surface of the specimen, the signal from the sputtered ions or secondary electrons can be collected to form an image of the surface of the sample. FIB systems can be operated at low beam currents for imaging or at high beam currents for site specific sputtering or milling. At low beam currents, very little material is sputtered and modern FIB systems can easily achieve 5 nm imaging resolution (Orloff et al., 1996). At higher currents, big amounts of material can be removed by sputtering, allowing precision milling of the specimen down to submicrometer or even tens of nanometer resolution. Additionally, gas delivery systems can be used in conjunction with the ion beam to produce site specific deposition of metals or insulators or to provide enhanced etching capabilities.

In this work in a FEI Helios NanoLab 600i FIB FEG-SEM dual-beam microscope was used for site-specific material removal.

Fig. 3.9 (a) FEI Helios NanoLab dual-beam FIB-SEM microscope utilised in the present thesis; (b) interior of the vacuum chamber; (c) schematic representation of the different components. Figure by Schiffbauer and Xiao.

3.4.1 Micropillar fabrication

Micropillars (MPs) with dimensions of $5 \times 5 \times 10 \text{ } \mu\text{m}^3$ were fabricated out of the colonies of interest, with lamellar orientations (Φ_L) close to 0° , 45° and 90° with respect to the loading axis. These three types of orientations were expected to exhibit well-differentiated mechanisms of deformation and, consequently, different mechanical responses. Since the inspection of the specimens in the SEM can only reveal the traces of the lamellae on the surface, the selection of the colonies was conducted by milling cross-sections of individual colonies with the FIB prior to the fabrication of MPs. The stage of the microscope was first tilted 52° to set the polished surface of the specimen perpendicular to the ion beam. Then, trenches were milled in several colonies perpendicularly to the lamellar traces visible on the surface of the specimens and the lamellar orientation was measured with the SE signal. Colonies with lamellae forming angles close to 0° , 45° and 90° were then selected for MP fabrication. Subsequently, two to four MPs were machined within each colony to gain a better statistical representation of the mechanical response of the colonies under study. To ensure reproducibility of the MPs fabricated, a milling pattern was designed with a square spiral path that allowed the beam to raster the four sides of the pillar in the

same direction, avoiding the asymmetries produced at the top edges. Moreover, the whole process was automated through the iFast software, provided by FEI, that automatically sets the programmed ion beam conditions and positions the milling patterns according to edge detection tools based on SE image contrast. The whole milling process can be divided in the following six steps (see Figure 3.10): (i) manual positioning of the sample so that the lamellar traces are parallel to one side of the MP; (ii) rough milling with 21 nA current for fast removal of material; (iii) milling with 9.3 nA current to approach the final dimensions of the MP; (iv) milling with 2.5 nA current down to the final dimensions of the MP; (v) removal of taper by cleaning two sides of the sample using a current of 0.23 nA nA, after positioning the stage at a tilt of -9° ; and (vi) 90° rotation of the sample to remove taper, repeating step (v).

Fig. 3.10 SEM micrographs illustrating the micropillar milling procedure.

3.4.2 TEM lamella fabrication

Because of the advantages that FIB offers for site-specific material removal, these systems have emerged as a powerful tool for TEM specimen preparation (Lucille and Fred). Preparation of electron transparent foils out of the deformed specimens was carried out following the lift-out technique, as illustrated in Figure 3.11. The steps for extracting a TEM foil are the

Fig. 3.11 Steps for TEM foil extraction (adapted from <https://www.pdx.edu/cemn/fib-gallery>).

following: (i) selection of the area of interest; (ii) deposition of a Pt protective layer to avoid Ga^+ ions impinging the surface from introducing damage; (iii) rough milling of trenches to remove the surrounding material with currents ranging from 9.3 to 21 nA, leaving a thin wall of about 2 μm thickness; (iv) thinning of the wall with lower beam currents (0.79 nA), to avoid damage of the sample; (v) milling of frame cuts positioning the stage at 45° or 60° , to leave the membrane attached to the bulk by a corner of it (0.79 to 2.5 nA); (vi) polishing with lower beam currents to the near nominal thickness (40 to 80 pA); (vii) attachment of the foil to a micromanipulator by depositing Pt; (viii) final milling to free the membrane; (ix) attachment of the membrane to the TEM grid by depositing Pt and (x) milling to free the membrane from the micromanipulator (0.79 to 2.5 nA).

3.5 Mechanical testing by instrumented nanoindentation systems

Microcompression experiments are usually carried out using instrumented nanoindentation systems, adapted by substituting the regular indenter tip with a customised flat-end tip (flat punch). Nanoindentation systems have been extensively used for the mechanical characterisation of materials since the mid-1970s (Bulychev et al., 1975; Loubet et al., 1984; Newey et al., 1982; Pethica et al., 1983; Poon et al., 2008). Their capabilities have evolved along the years to include, besides the measurement of hardness and elastic modulus (Bhushan, 1999; Oliver and Pharr, 2004, 1992) in small volume samples, the possibility to probe of other mechanical parameters, such as hardening exponents (Bucaille et al., 2003; Chollacoop et al., 2003; Dao et al., 2001; Kucharski and Mróz, 2001; Ma et al., 2003), creep properties (Fischer-Cripps, 2004; Lucas and Oliver, 1999; Oyen and Cook, 2003; Storåkers and Larsson, 1994; Takagi et al., 2003) and residual stresses (Carlsson and Larsson, 2001a,b; Suresh and Giannakopoulos, 1998; Swadener et al., 2001), as well as high temperature capabilities and *in situ* imaging of the indented volume (Schuh, 2006). Nowadays, owing to the development of new sensors and actuators, the systems used for nanoindentation testing are capable of measuring contact forces and displacements in the nN and nm range, respectively, and are furnished with positioning resolution below 1 μm . This combination of their capabilities has made nanoindentation a preferred technique for the mechanical characterisation at sub-micron scales.

A nanoindentation system is basically made of an indenter, as well as sensors and actuators used for controlling the applied force, measuring the tip displacement, and positioning the

stage. If the regular indenter tip is substituted by a flat-end tip, or flat punch, the capabilities of these systems can be extended to micro-compression experiments (see reviews (Greer and De Hosson, 2011; Kraft et al., 2010; Uchic et al.)). These tests mimic the compression tests commonly performed on macroscopic samples, with modifications to facilitate the fabrication and manipulation of the small specimens (Uchic et al., 2004; Uchic and Dimiduk, 2005; Uchic et al., 2003). The most significant difference between macro- and microtests is that, in the latter, the samples are not freestanding; instead, they remain integrally attached to the bulk substrate to eliminate the need for micromanipulation. Hence, the substrate acts as the lower compression plate during the test, and its stiffness contributes to the measured displacement. This must be carefully taken into account if accurate modulus and strain values are to be measured. Additionally, misalignment between the indenter axis and the micropillar should be kept to a minimum, since it could lead to an underestimation of the yield point and the elastic modulus, to changes in the strain-hardening response and to buckling (Choi et al., 2007; Ng and Ngan, 2008), especially when they are greater than 1° . Several numerical and analytical methods have been proposed to account for the substrate deflection (Choi et al., 2007; Greer et al.; Zhang et al., 2006). In this thesis, the procedure described by Sneddon for a perfectly rigid flat punch indenting into an isotropic elastic half space has been utilised. This method is explained in detail in Chapter 4.

In this work, two types of nanoindentation systems have been used: an *ex situ* Hysitron TriboIndenter® TI 950 (Figure 3.12a), and an *in situ* Hysitron TriboIndenter® PI 87 (Figure 3.12b), whose functioning is described as follows.

Fig. 3.12 (a) Hysitron TriboIndenter® TI 950 and (b) PI 87.

***Ex situ* nanoindentation system**

The *ex situ* experiments were carried out in a commercial TriboIndenter® TI 950 nanoindentation system from Hysitron, Inc. (Minneapolis, MN) with an environmentally dedicated enclosure where samples are thermally and acoustically isolated. This system is a self-contained, stand-alone platform, intrinsically load-displacement controlled. The force is applied in the vertical direction, electrostatically, and the displacement is measured by utilising a patented design of a three-plate capacitive transducer. The low-load transducer offers a maximum force of 12 mN with a force resolution smaller than 1 nN and a displacement resolution below 0.02 nm; the high-load transducer presents a maximum force of 1 N with a force resolution smaller than 1 μ N and a displacement resolution below 0.1 nm. The nanoindentation system set up is shown in Figure 3.12a.

***In situ* nanoindentation system**

In situ micropillar compression and tensile tests were carried out inside the FEI Helios NanoLab 600i FIB FEG-SEM dual beam microscope using a commercial TriboIndenter® PI87 nanoindentation system from Hysitron, Inc. (Minneapolis, MN). The *in situ* nanoindentation system is a miniaturised test platform for *in situ* materials characterisation at variable temperatures. This system is a depth-sensing mechanical test instrument designed with a vacuum compatible transducer and an electrically conductive probe to perform quantitative nanomechanical testing while simultaneously imaging with the SEM. In this system, a force is applied electrostatically and the displacement is measured capacitively. This low-current design provides low thermal drift and a high stability and sensitivity. Two transducers are available: the low-load transducer has a maximum force of 12 mN with a force resolution smaller than 1 nN and a displacement resolution below 0.02 nm; the high-load transducer presents a maximum force of 150 mN with a force resolution below 1 μ N and displacement resolution below 0.1 nm. The specimen is mounted on a piezo-actuated stage with 5 degrees of freedom, which allows accurate alignment between the load head and the specimen, as well as its optimum positioning for imaging with the different detectors. Furthermore, this system has the capability to conduct testing at high temperatures by incorporating tip and sample heaters with independent thermocouple-controlled feedback loops up to 800 °C. The system set up is shown in Figure 3.12b.

Chapter 4

Effect of lamellar orientation

The objective of the work presented in this chapter is to determine the influence of lamellar orientation on the strength and operative deformation mechanisms of a fully lamellar Ti-45Al-2Nb-2Mn (at.%) + 0.8(vol.%) TiB₂ (Ti4522XD) alloy. With this aim, micropillars with lamellae oriented at 0°, 45° and 90° with respect to the loading direction were compressed at room temperature. The results revealed a large plastic anisotropy, that was rationalised, based on slip/twinning trace analysis, according to the relative orientation of the main operative deformation modes with respect to the lamellar interfaces. Loading at 45° resulted in the activation of soft longitudinal modes, where both the slip plane and the slip direction were parallel to the interfaces, and therefore, little interaction of dislocations with lamellar interfaces is expected. At 0° loading, deformation was mainly accommodated by harder mixed deformation modes (with an oblique slip plane but a slip direction parallel to the lamellar interfaces), although the lamellar interfaces seemed to be relatively transparent to slip transfer. On the contrary, 90° loading represented the hardest mode and deformation was accommodated by the activation of transverse deformation systems, confined to individual lamellae, together with longitudinal modes that were activated due to their softer nature, despite their very small Schmid factors. A thorough study of pillar size effects revealed that the results were insensitive to pillar size for dimensions above 5 μm . The results can therefore be successfully applied for developing mesoscale plasticity models that capture the micromechanics of fully lamellar TiAl microstructures at larger length scales.

4.1 Introduction

Due to the intricate nature of the polycrystalline FL microstructures, governed by a large number of parameters (composition, colony size, lamella size, volume fraction of the two phases, misorientation and character of the interfaces), quantifying the effect of each microstructural feature on the overall deformation and fracture behaviour of polycrystalline γ -TiAl specimens is, thus, extremely challenging (Muñoz-Moreno et al., c, 2012). It is crucial to gain a better understanding of the fundamentals of crystallographic slip and twinning in this material, and their interaction with the characteristic lamellar microstructure. Broadening current knowledge in this topic would constitute a valuable tool for enhancing alloy development. Furthermore, with the aim of developing numerical models capable of reproducing the behaviour of fully lamellar microstructures, statistically sound experimental data regarding the influence of phase distribution, crystallographic orientation and lamellar interface nature on slip activation is required. Such studies are currently scarce (Lebensohn et al.; Zambaldi et al., a).

The aim of the work presented in this chapter is to characterise the influence of lamellar orientation with respect to the applied load on the active deformation modes in individual colonies of a centrifugally cast FL Ti-45Al-2Nb-2Mn (at.%) + 0.8(vol.%) TiB₂ (Ti4522XD) alloy. With the aim of seeding light on the mechanisms of deformation acting at the microscale in the alloy under study, micropillar compression tests were conducted at room temperature. This methodology allowed us to elude the complexity of the microstructure that may influence the plastic deformation at different scales in a polycrystalline bulk material, thus, providing a better understanding of the influence that the lamellar orientation has on the deformation behaviour of individual colonies. Moreover, in contrast to previous single crystal and PST studies, the experimental methodology presented in this work is well suited to study the behaviour of polycrystalline γ -TiAl alloys of industrial interest at the microscale level. As a great number of micropillars can be taken from a same bulk specimen, a good statistical representation of the effect of the lamellar orientation may be achieved with no need of producing a large number of bulk specimens that would require a costly and time-consuming manufacturing process.

4.2 Experimental

Prior to micropillar fabrication, the specimen surface was prepared following Section 3.2. The micropillars were machined by FIB milling in colonies with different lamellar orientations with respect to the compression axis, Φ_L . In particular, values of 0° , 45° and 90° were selected as they were expected to exhibit well-differentiated mechanisms of deformation (Inui et al., 1992b). The micropillars were machined following the procedure described in Section 3.4.1 with a square cross-section to facilitate the measurement of the slip traces. Pillars with side dimensions in the range between 1 to $7\mu\text{m}$ were tested in order to make sure that the measured flow stress were free of pillar size effects and were thus representative of the bulk behaviour. A height to side dimension ratio of 2 : 1 was selected in all cases to avoid buckling effects (Figure 4.1).

Fig. 4.1 High resolution SEM micrograph of a micropillar with square cross section.

Uniaxial compression of the micropillars was carried out at room temperature using a TI 950 TriboindenterTM (Hysitron, INC, Minneapolis, MN). The samples were fixed to a magnetic holder by means of mechanical clamps seeking for a stiff assembly during testing. A flat-punch diamond tip with a diameter of $10\mu\text{m}$ was installed on a high-load transducer to apply the force. The tests were carried out in a displacement control mode at a strain rate of 10^{-3}s^{-3} up to a maximum strain of 10%. The load-displacement curves were corrected for the elastic deflection of the substrate following the Sneddon correction (Greer et al.; Sneddon) and converted to engineering stress-strain based on the initial cross-sectional area and length

measured after the milling process. Finally, the curves were transformed to engineering stress-plastic strain by subtracting the elastic strain from the elastic modulus measured at the final elastic unloading. It is well known that, due to local surface irregularities and/or small misalignments between the flat indenter tip and the pillar head, the initial loading is more compliant than what one would expect from a purely elastic loading (Soler et al., a). In the current case, however, the initial plastic yield was clearly marked by a distinct displacement burst in all cases, as indicated by a black arrow in Figure 4.2. Based on the definition of the yield point, the engineering stress-plastic strain curves could then be corrected by shifting the plastic strain at the yield point to the origin. Figure 4.2 shows the comparison between the raw engineering stress-strain curve for a micropillar with $\Phi_L = 90^\circ$. The non-linearity at the last stages of the unloading segment is associated with Bauschinger effects.

Fig. 4.2 Representative stress-strain curve before and after data correction corresponding to a MP with $\Phi_L = 90^\circ$. The black arrow indicates the yield point. Note that the non-linearity of the unloading curve might be associated to Bauschinger effects.

High resolution scanning electron microscopy (HR-SEM) was used to analyse the deformed pillars and to measure the geometry of the slip traces that appeared on the free surfaces of the pillar upon deformation. This analysis is crucial for two reasons. First, by relating the slip plane and slip direction to the orientation of the lamellar interfaces, the slip traces could be assigned to the different deformation modes (longitudinal, mixed, transversal), according

to the morphological classification described in the introduction section. And second, by estimating the Schmid factors with respect to the applied force, the applied resolved shear stress could be calculated during deformation, in order to estimate the critical resolved shear stress (τ_{CRSS}) for each deformation mode. The analysis of the geometry of the slip traces was facilitated in this case by the use of square pillars, so that the orientation of the slip plane and slip direction could be readily determined from the slip traces detected on the flat surfaces of the pillars. The process is illustrated in Figure 4.3. Attending solely to geometrical considerations, the τ_{CRSS} of a given slip system can be obtained from expression 4.1:

$$\tau_{CRSS} = \sigma_y \cdot m \quad (4.1)$$

where σ_y is the measured yield stress, as indicated in Figure 4.3, and m the Schmid factor associated to the operative slip system. m is given by Equation 4.2, where ϕ represents the angle between the slip plane normal and the loading direction and λ is the angle between the slip and loading direction.

$$m = \cos(\phi) \cdot \cos(\lambda) \quad (4.2)$$

The angles ϕ and λ can be directly obtained from slip line analysis using expressions 4.3 and 4.4:

$$\phi = \arccos\left(\frac{\bar{l} \cdot \bar{n}}{\|\bar{l}\| \cdot \|\bar{n}\|}\right) \quad (4.3)$$

$$\lambda = \arccos\left(\frac{\bar{l} \cdot \bar{d}}{\|\bar{l}\| \cdot \|\bar{d}\|}\right) \quad (4.4)$$

where $\bar{l}$, the loading direction, is equal to $-\bar{k}$ for the chosen coordinate reference system (Figure 4.3); $\bar{n} = n_x\bar{i} + n_y\bar{j} + n_z\bar{k}$ is the slip plane normal; and $\bar{d}$ is the slip direction. $\bar{n}$ and $\bar{d}$ can be calculated through expressions 4.5 and 4.6, respectively:

$$\bar{n} = \bar{n}_1 \times \bar{n}_2 \quad (4.5)$$

$$\bar{d} = a\bar{i} + b\bar{j} + c\bar{k} \quad (4.6)$$

where $\bar{n}_1 = -\cos(\alpha_1)\bar{i} + \sin(\alpha_1)\bar{k}$ and $\bar{n}_2 = -\cos(\alpha_2)\bar{j} + \sin(\alpha_2)\bar{k}$ are unit vectors parallel to the slip traces observed among two mutually perpendicular surfaces of the micropillar (Figure 4.3). a and b in expression 4.6 can be directly measured from the relative displacements in the x and y directions, respectively, of the slipped part of the pillar over the undeformed part, by imaging the pillar along the top view. For measuring c , the displacement in the z

direction, one must consider that $\bar{d}$ is contained in the slip plane and hence $\bar{d} \cdot \bar{n} = 0$, from which:

$$c = -\frac{a \cdot n_x + b \cdot n_y}{n_z} \quad (4.7)$$

This methodology was easily applied to pillars deformed with $\Phi_L \approx 0^\circ$ and 45° . However, for pillars with $\Phi_L \approx 90^\circ$, i.e., with lamellar orientation perpendicular to the loading axis, the determination of the slip direction from the top view of the deformed pillar was difficult because deformation was considerably constrained. In this particular case, an upper bound for the τ_{CRSS} was estimated by considering the slip direction to be coplanar with the loading axis and the normal to the slip plane, i.e., $\cos(\lambda) = \cos(\lambda)|_{max}$.

Fig. 4.3 Illustrative representation of trace analysis in a deformed MP. Red lines represent traces associated to the operative system of deformation, $\bar{n}$ and $\bar{d}$ stand for the normal to the plane and direction of deformation, respectively; and a , b , and c correspond to the measured displacement of the deformed part of the MP in the coordinate system indicated in the figure.

Finally, the deformation mechanisms were further analysed by transmission electron microscopy at 200 kV in order to elucidate the mechanisms involved for each deformation

mode. For this, electron transparent foils were milled along selected deformed pillars in the FIB.

4.3 Results and discussion

A size-effect study was initially conducted to validate the MP dimensions that best represent the behaviour of single colonies of the γ -TiAl alloy under study, thus avoiding strengthening effects associated with the dimensions of the samples. Figure 4.4 illustrates the variation of the yield stress as a function of the MP diameter. Yield values, σ_y , remain constant for pillars with diameters larger than $5\mu\text{m}$ and increases abruptly for thinner pillars. Based on these results, MPs with a diameter of $5\mu\text{m}$ were considered to be free of size effects.

Fig. 4.4 Size effects in a Ti4522XD alloy as function of Φ_L .

4.3.1 Deformation mechanisms

4.3.1.1 Soft mode ($\Phi_L = 45^\circ$)

MPs with lamellae orientation corresponding to the soft mode were deformed up to a strain of 10%. Two representative stress-strain curves are plotted in Figure 4.5, following the

Fig. 4.5 Experimental engineering stress-strain curves corresponding to $\Phi_L = 45^\circ$. Colored diamonds indicate initiation of yielding.

correction procedure described in Section 4.2. The scatter between different curves was not large, indicating that local microstructural variations from pillar to pillar (like the actual lamellae thickness distribution) are not affecting strongly the results. The initiation of plastic deformation (yield stress), associated to the first large burst in the recorded data, is indicated with diamonds. The yield stress and the ultimate stress measured for this condition are, respectively 682 ± 38 MPa and 884 ± 137 MPa. Losses of contact between the indenter and the sample, revealed by the discontinuities apparent in the curves and originated by abrupt sample downwards displacements, occur repeatedly during the test. More specifically, displacements ranging from 2 to 359 nm are observed, with an average value of 12 ± 44 nm. Stress drops in nanoindentation curves are often attributed to either dislocation avalanches and/or to the operation of twinning (Ng and Ngan, 2008). In order to further elucidate the operative deformation modes, the surfaces of the deformed MPs were carefully examined by FEG-SEM. Figure 4.6a-b illustrate the compressed MPs corresponding to the curves of Figure 4.5. the presence of traces parallel to the lamellar interfaces is clearly consistent with the preferred activation of longitudinal soft deformation modes. A closer examination of the surfaces reveals the presence of both slip and twinning events. The image of the MP in Figure

4.6a shows, first, sharp discontinuities in the surfaces (black arrows), which correspond to slip steps. Second, lamellae deflection at the edges of the MPs (white arrows), suggesting the presence of twinning, is also apparent. Finally, some degree of interfacial sliding (red arrow), possibly induced by the high stress levels attained close to the are of contact, also appears to be present. Deformation in the second MP, imaged in Figure 4.6b, appears to be dominated by slip, as the surfaces are populated with steps and little evidence of lamellae deflection is present. However, the resolution of FEG-SEM imaging does not allow for a clear distinction between very thin twins and slip traces. In order to gain further insight into the operative deformation modes, TEM foils were extracted perpendicular to the lamellae interfaces. BF images confirmed the presence of deflections on the sides of the MPs (Figure 4.6c-d), likely emerging from the high shear strains associated to strain accommodation by longitudinal twinning in the absence of mechanical constrains. The contrast apparent within lamellae is also consistent with the occurrence of dislocation activity.

Fig. 4.6 (a,b) FEG-SEM micrographs illustrating the surfaces of micropillars compressed in the soft mode $\Phi_L = 45^\circ$. The dashed lines highlight the lamellae orientation. Traces of longitudinal slip (black arrows), twinning (white arrows) and interface shear (red arrows) are observed; (c,d) BF-TEM images of samples extracted from the MP in (a).

An analytical procedure was utilised in order to estimate the number of dislocations that would cause the stress drops observed in Figure 4.5. Figure 4.7a illustrates a cross-section of a compressed MP with a single surface step that, in this ideal case, is entirely attributed to slip activity. The number of dislocations leading to the formation of such a step may be calculated by dividing the displacement along the longitudinal plane, s , by the corresponding Burgers vector. This calculation revealed that the minimum measured downward displacement, 2 nm, would be equivalent to avalanches of 10 ordinary dislocations or 5 superdislocations; also, the maximum measured downward displacement, 359 nm, would be equivalent to avalanches of 1795 ordinary dislocations or 889 superdislocations. These values seem to agree reasonably well with dislocation activities quantified in earlier studies (Ng and Ngan, 2008).

Fig. 4.7 Illustrative representation of a step emerging at the side of a MP when longitudinal slip occurs (a) and deflection of the side of MP due to longitudinal twinning (b).

A second analytical procedure, described in Figure 4.8, was utilised in order to estimate the thickness of a twin (Figure 4.7b) whose formation could potentially have caused the stress drops observed in Figure 4.5. Figure 4.8a represents the normal projection of two twin related planes where the reference directions are indicated for both planes with red

arrows in opposite sense; Figure 4.8b illustrates the $(111)_\gamma$ twin plane (black dotted line) and the $[11\bar{2}]_\gamma$ twin direction (red dotted line) in the fct lattice. A cross section of Figure 4.8b was then sketched in Figure 4.8c taking into account that the parent and twin regions would share a $(111)_\gamma$ plane while having $[\bar{1}\bar{1}0]_\gamma$ directions in opposite senses. As can be seen in Figure 4.8d, the twin thickness (t) corresponding to a downward displacement l_1 , might be calculated from the corresponding surface relief angle (φ_r) using the following equations dragged from Figure 4.8f:

$$\varphi_r = \frac{\pi}{2} - \Phi_L - \arctan\left(\frac{2c}{d}\right) \quad (4.8)$$

$$l = \frac{t}{\sin(\Phi_L)} \quad (4.9)$$

$$l = l_1 \cdot \left(1 + \frac{\tan(\Phi_L)}{\tan(\varphi_r)}\right) \quad (4.10)$$

$$t = l_1 \cdot \left(1 + \frac{\tan(\Phi_L)}{\tan(\varphi_r)}\right) \cdot \sin(\Phi_L) \quad (4.11)$$

Using this method, the t values calculated ranged from 10 nm (downward displacement of 2 nm) to 1700 nm (downward displacement of 359 nm). Again, these values seem of the order of the lamellae thicknesses ($\lambda_{\alpha_2} = 319 \pm 339$ nm and $\lambda_\gamma = 287 \pm 133$ nm) and, thus, would further support the idea that the stress drops observed could be attributed to longitudinal twinning events.

Despite the impossibility of distinguishing the frequency of twinning and slip events both by experimental and analytical techniques, our results clearly reveal that deformation up to 10% in the compressed MPs is accommodated mainly by longitudinal deformation modes. The trace analysis method described in Section 4.2 was utilised to calculate an average value for the critical stress necessary to promote plastic deformation along longitudinal systems in MPs with lamellar orientations close to 45° . The value obtained was 209 ± 68 MPa. Previous studies have confirmed the preferential activation of longitudinal slip and twinning during compression up to strains of 5% (Inui et al., b, 1992b) in PST samples and of twinning in MPs (Edwards et al.) loaded in soft orientations. However, other works (Edwards et al.) have reported a relatively high activity of out-of-plane deformation above 5% strain. The apparent discrepancies between our work and the literature regarding the out-of-plane activity at strains higher than 5% might potentially derive from the different constraints imposed by our experimental set-up and that of earlier works. For example, it is likely that the lateral stiffness of the grips of a conventional testing machine imposes large constraints on

Fig. 4.8 Geometrical aspects related to mechanical twinning in an ideal γ single crystal MP with the $(111)_\gamma$ planes oriented at 45° . (a) Normal projection of two $(111)_\gamma$ twin related planes where the reference directions $[\bar{1}\bar{1}0]_\gamma$ are indicated for both planes with red arrows in opposite sense; (b) unit cell with lattice parameters a and c , and diagonal of the basal plane d . The $(111)_\gamma$ twin plane (black dotted line) and the $[11\bar{2}]_\gamma$ twin direction (red dotted line) are drawn in the fct lattice; (c) projection along the $[\bar{1}\bar{1}0]_\gamma$ direction where the shared $(111)_\gamma$ trace for a twinned misorientation relationship between two crystals is observed, (d) schematic illustrating how a reference single gamma crystal would look like if deformed through longitudinal twinning. The downwards displacement, l_1 , indicated in (e), is determined assuming constant volume of the twinned region before and after deformation. t represents the thickness of a twinned lamella after deformation of the MP. Geometrical details of the analysis are given in (f).

macroscopic PST samples during compression, severely hindering longitudinal shear for large strains, and originating the higher stress values that are required for the activation of out-of-plane deformation modes.

4.3.1.2 Hard mode I ($\Phi_L = 0^\circ$)

MPs with lamellar orientation corresponding to the hard mode I were deformed up to a strain of 10%. Two representative stress-strain curves are plotted in Figure 4.9, following the correction procedure described in Section 4.2. The initiation of plastic deformation (yield stress), associated to the first large burst in the recorded data, is indicated with diamonds. The yield stress and the ultimate stress measured for this condition are, respectively, 1096 ± 22 MPa and 1443 ± 180 MPa.

Fig. 4.9 Experimental engineering stress-strain curves corresponding to $\Phi_L = 0^\circ$. Colored diamonds indicate initiation of yielding.

In order to further elucidate the operative deformation modes, the surfaces of one of the deformed MPs were carefully examined by FEG-SEM, specifically the one corresponding to the red curve. Fig 4.10a-c illustrate two perpendicular surfaces of the same compressed MP. Since lamellae could not be clearly distinguished in the images, their orientation is indicated in Figure 4.10a-g, using white lines. Figure 4.10a reveals the absence of any lateral

Fig. 4.10 (a-c) FEG-SEM micrographs of perpendicular surfaces corresponding to a compressed MP with lamellar orientation $\Phi_L = 0^\circ$. White dashed lines highlight the lamellae orientation. White arrows in (a) and (b) represent the projection of the slip direction on the top surface of the pillar, parallel to the lamellar interfaces; (d) BF-TEM micrograph showing twinning activity within lamellae at the bottom of the specimen; (e) BF-TEM micrograph illustrating localised cracking close to the contact surface of the MP; (f) Transverse twinning across lamellae located close to the base of the MP.

displacement perpendicular to the lamellae. Thus, it can be unambiguously concluded that the slip direction is parallel to the lamellae, as indicated by the white arrow in Figure 4.10a and Figure 4.10b. Figure 4.10c reveals, further, that deformation is preferentially accommodated by mixed slip systems, as slip traces make relatively high angles with respect to the lamellar interfaces. The hardening values observed in the experimental blue curve in Figure 4.9 might thus be a consequence of cross-hardening between different mixed systems presenting similar m values (indicated with white dashed lines on the side of the MP in Figure 4.10b). A closer inspection of the interior of the lamellae by BF-TEM confirms the presence of a high dislocation activity (Figure 4.10f). The preferential activation of mixed systems in MPs with $\Phi_L = 0^\circ$ is to be expected, as longitudinal slip is prevented due to geometrical considerations (very low m) and transverse systems, which would also have relatively high m values, are not able to accommodate as much strain due to the presence of a larger number of interfaces along the dislocation slip path. The trace analysis method described in Section 4.2 was utilised to calculate an average value for the critical stress necessary to promote plastic deformation along mixed systems in MPs with lamellar orientations close to 0° . The value obtained was 458 ± 9 MPa.

Fracture of lamellae is occasionally observed in some regions close to the zone of contact (Figure 4.10e). Finally, localised transversal twinning was found to be operative in some adjacent γ variants located close to the base of the MP (Figure 4.10d) due to the constraint that exists at the bottom of the pillar, in agreement with results reported in previous work (Rester et al.).

4.3.1.3 Hard mode II ($\Phi_L = 90^\circ$)

MPs with lamellae orientation corresponding to the hard mode II were deformed up to a strain of 10%. Two representative stress-strain curves are plotted in Figure 4.11, following the correction procedure described in Section 4.2. The initiation of plastic deformation (yield stress), associated to the first large burst in the recorded data, is indicated with diamonds. The yield stress and the ultimate stress measured for this condition are, respectively, 1410 ± 76 MPa and 1781 ± 76 MPa. Non-negligible hardening levels are observed, together with the presence of stress bursts which are, however, less frequent than in MPs with $\Phi_L = 45^\circ$.

Previous studies reported high activity of transversal twinning for this loading mode (Inui et al., a; Rester et al.). Longitudinal and mixed activity should be inhibited due to zero m values. However, in this study it was experimentally impossible to fabricate MPs

Fig. 4.11 Experimental engineering stress-strain curves corresponding to $\Phi_L = 90^\circ$. Colored diamonds indicate initiation of yielding.

with lamellae at exactly right angles with respect to the loading direction. Figure 4.12a and b are FEG-SEM micrographs that illustrate the lateral surfaces of two undeformed MPs with lamellae quasi-perpendicular to the loading axis. The measured deviation angle ranged between 7° to 12° , thus leading to nonzero resolved shear stress (τ) values associated to longitudinal systems. Figures 4.12c and d are FEG-SEM micrographs corresponding to the compressed MPs. Traces associated to longitudinal and transverse activity are highlighted with white and black arrows, respectively. Longitudinal slip seems, indeed, predominant. The trace analysis method described in Section 4.2, particularised for this loading mode, was utilised to calculate an average value for the critical stress necessary to promote plastic deformation along longitudinal systems in MPs with lamellar orientations between 83° and 78° . The value obtained was 217 ± 12 MPa.

Figures 4.12e and f are BF-TEM images of a foil that was extracted parallel to a side of one of the deformed MPs. These images reveal the presence of thin plates transverse to lamellar boundaries, which are characteristic transverse twinning. The SADP ($[\bar{1}10]_\gamma$ zone axis) in Figure 4.12g was obtained from a region populated with the described traces. Pattern indexing (Figure 4.12h) confirms a twin misorientation relationship between the

Fig. 4.12 HR SEM micrographs of a MP with $\Phi_L = 90^\circ$ (a,b) before compression and (c,d) after compression. White arrows indicate longitudinal activity while black arrows correspond to transversal traces. (e,f) BF-TEM micrograph of a nanotwinned region. (g) SADP of the matrix and its twin with a common $\langle \bar{1}10 \rangle_\gamma$ zone axes; (h) indexed pattern labeled as M (matrix) and T (twin), with a common $(\bar{1}\bar{1}1)_\gamma$ reflection.

matrix and the thin plates (rotation of 180° around the $[\bar{1}\bar{1}1]_\gamma$ common axis). The weakest spots correspond to the small twinned volumes. The transverse nature of this nano twin mechanism of deformation makes it difficult to accomplish large strain values, as is restricted to a very small volume fraction of the specimen. The critical stress necessary to promote plasticity along this transverse twinning system, with a measured m of 0.44, was found to be 620 ± 33 MPa.

Fig. 4.13 Plastic anisotropy observed at room temperature. Data corresponding to the Ti4522XD alloy (present Chapter), Ti-49.3Al (Fujiwara et al., 1990), Ti-45Al-8Nb (Peng et al.) and Ti-48Al (Kim et al., 1999b) alloys.

In summary, deformation in MPs with lamellae that are quasi perpendicular to the loading axis, longitudinal slip appears to accommodate most of the strain imposed by the flat punch; the characteristic low τ_{CRSS} associated to these systems make them prone to activation even for low m values. Thus, the high strength of the specimens tested at orientations close to 90° should result from these very low Schmid factor longitudinal systems governing the deformation of the pillars. Additionally, minor contributions of transverse twinning are also observed. The strain bursts observed in the stress-strain curves of Figure 4.11 may, thus, be attributed to avalanches of dislocations or twinning events on longitudinal systems. The fact that these bursts are less frequent than those occurring under soft loading conditions may be

explained by the significantly smaller m values of longitudinal systems in the hard loading mode ($m = 0.15$).

Table 4.1 summarises the dominant deformation systems, the yield and ultimate stresses, as well as the τ_{CRSS} values corresponding to the three loading modes analyzed. Furthermore, Figure 4.13 compares the current yield stress results with those obtained by other authors from PST studies in several TiAl alloys (Fujiwara et al., 1990; Kim et al., 1999b; Peng et al.). Unfortunately, there are no studies in PST of the same composition, but the values determined capture well the plastic anisotropy observed in other TiAl alloys and lie in between those found for Nb-free TiAl alloys and alloys with a higher content of Nb, and therefore, rendering the quantitative results reasonable.

Table 4.1 Dominant deformation mode, yield stress and ultimate strength, together with the calculated initial τ_{CRSS} , as a function of loading angle.

$\Phi_L(^{\circ})$	Dominating deformation mode	Yield stress (MPa)	Ultimate strength (MPa)	τ_{CRSS} (MPa)
0	Mixed	1096 ± 22	1443 ± 180	458 ± 30
45	Longitudinal	682 ± 171	884 ± 137	209 ± 68
90	Longitudinal	1410 ± 76	1781 ± 76	217 ± 12
90	Transverse			620 ± 33

4.3.2 Analysis of size effects

As shown in Figure 4.4, substantial pillar size effects were found for pillar sizes below $5\text{ }\mu\text{m}$. A simplified attempt to rationalise the trends observed is carried out in view of the widely accepted theory of size effects developed for single crystal micropillars in the last ten years (Dehm; Greer and De Hosson, 2011; Parthasarathy et al.; Rao et al.; Tang et al.; Uchic et al.). Pillar size effects in single crystals are thought to arise due to the operation of single arm dislocation sources between an internal pinning point and the pillar surface (Parthasarathy et al.). Following Parthasarathy, the critical resolved shear stress τ_{CRSS} for the activation of a single arm dislocation source can be calculated by adding the bulk strength of the single crystal τ_{bulk} to the shear stress required to activate single arm sources τ_{λ_S} :

$$\tau_{CRSS} = \tau_{bulk} + \frac{\alpha\mu b}{\bar{\lambda}_{max}(D, \rho_{tot}, \beta)} \quad (4.12)$$

where τ_{bulk} represents the bulk strength of the single crystal, μ the anisotropic shear modulus, b the Burgers vector, α a constant in the order of unity and $\bar{\lambda}_{max}$ the statistical average length of the weakest single arm dislocation source, which is a function of the micropillar diameter, D , the dislocation density, ρ_{tot} , and the slip plane orientation, β . It follows that the relative weight of each term in Equation 4.12 is responsible for the different levels of size effects observed in different types of materials or at different temperatures (Korte and Clegg; Soler et al., b).

The case of micropillars of FL γ -TiAl alloys is far more complex, as a competition between several characteristics length scales is established: an extrinsic length-scale, associated with the side length of the pillar, and hence, the size of the operating dislocation source, λ_S , and an intrinsic one, characterised by the characteristic size of the microstructure, which in this case is represented by the colony size, λ_C , the domain size, λ_D , and the lamellar thickness, λ_L . Therefore, assuming the same simple additive approach as that represented by Equation (4.12), this can be rewritten for the case of FL γ -TiAl, as:

$$\tau_{CRSS} = \tau_{TiAl} + \tau_{\lambda_C} + \tau_{\lambda_D} + \tau_{\lambda_L} + \tau_{\lambda_S} \quad (4.13)$$

where τ_{TiAl} stands for the lattice resistance of the γ -TiAl crystal and τ_{λ_C} , τ_{λ_D} , τ_{λ_L} and τ_{λ_S} represent the strengthening associated to the colony size, the domain width, the lamellar thickness and the pillar size respectively. Considering that the pillars were fabricated out of single colonies, the colony size is, in this case, irrelevant. Regarding domain boundaries, those where typically spaced on average larger distances than the pillar diameter, so their contribution is expected to be negligible. Actually, domain boundaries are, in general very coherent (Appel et al., b). Therefore, the main contribution to the measured strength is expected to come from the inherent lattice resistance of the γ (TiAl) phase, and a competition between the intrinsic size effect associated with the lamellar thickness, τ_{λ_L} , and the extrinsic size effect, τ_{λ_S} , represented by the pillar size, and both should contribute differently depending on the operating deformation mode. In the following, a simple analysis of the micropillar compression results is performed in order to give estimates of each term in Equation 4.13.

First, it is instructive to compare the results of those loading orientations where only a single deformation mode is active. i.e., $\Phi_L = 45^\circ$ and $\Phi_L = 0^\circ$, representative of longitudinal and mixed deformation modes, respectively. Figure 4.14 plots the τ_{CRSS} determined as a function of MP diameter, for $\Phi_L = 0^\circ$ (Figure 4.14a) and $\Phi_L = 45^\circ$ (Figure 4.14b). For the longitudinal modes at $\Phi_L = 45^\circ$, slip takes place in planes parallel to the lamellae interfaces, and therefore, no dislocation-interface and/or twin-interface interactions are expected for this

Fig. 4.14 Size effects for MPs with lamellar orientations close to 0° and 45° . τ_{CRSS} calculated through trace analysis for mixed ($\Phi_L = 0^\circ$) and longitudinal systems ($\Phi_L = 45^\circ$).

soft deformation mode. As a result, for large pillars ($D > 5 \mu\text{m}$), the computed τ_{CRSS} is the lowest, at 209 MPa (blue dotted line in Figure 4.14b), which can be rendered as representative of the lattice resistance of the $\gamma(\text{TiAl})$ crystal, τ_{TiAl} , without contribution from the lamellar interfaces. Reference values for single-crystal $\gamma\text{-TiAl}$ specimens with comparable aluminum content by Inui et al. (1997) report a lower lattice resistance, of the order of 45 MPa. The higher value obtained in this case might be a consequence of two contributions: (i) the solid solution strengthening provided by Nb and Mn in the Ti4522XD alloy, and (ii) the strengthening arising from domain boundaries.

For the mixed modes at $\Phi_L = 0^\circ$, however, the slip planes are inclined with respect to the lamellar interfaces, while the slip direction remains parallel, and dislocations must necessarily interact with the lamellar interfaces, by either slip transfer across several lamellae or by bowing stress due to pinning of the dislocations. As a result, the computed τ_{CRSS} for $D > 5 \mu\text{m}$ increases up to 458 MPa (red dotted line in Figure 4.14a), and hence the difference with respect to the soft mode can be attributed to the interaction with the lamellar interfaces and the α_2 lamellae (Umakoshi et al.). The fact that the slip traces on the pillar surface are continuous across several lamellae (Figure 4.10c), but change their orientation, reflect that the γ/γ interfaces are relatively transparent to dislocations gliding on one lamella cross the interface to continue gliding on the slip plane with the most favoured Schmid factor on the adjacent lamella.

Interestingly, both loading directions show similar pillar size effects for $D < 3 \mu\text{m}$, as represented by the green dotted lines in Figure 4.14. In the present case, it was initially surprising that such pillar size effects could arise for pillars of several micrometers in size, containing both γ and α_2 lamellae, when the average lamellar thickness was only $\lambda_\gamma = 287 \pm 133 \text{ nm}$. However, the analysis of the deformation modes presented above demonstrate that similar dislocation surface interactions might arise for soft and mixed deformation modes in FL $\gamma\text{-TiAl}$. For soft deformation modes, $\Phi_L = 45^\circ$, with both the slip plane and slip direction parallel to the lamellar interfaces, a hypothetical source within a lamella would operate between an internal pinning point and the surface of the pillar, without interaction with the lamellar interfaces (see Figure 4.15a). For mixed deformation modes, $\Phi_L = 0^\circ$, for the smallest pillar sizes and considering the fraction of α_2 lamellae (31%), it is likely that most of the lamellae, if not all, are of the γ phase. The results demonstrate that the γ/γ interfaces are relatively transparent to dislocations with Burgers vectors parallel to the interface. As a result, it is not surprising that dislocations gliding across several lamellae

might interact with the pillar surfaces (see Figure 4.15b), even for pillar sizes larger than the average lamellar thickness.

Fig. 4.15 Operative single-arm dislocation source in soft mode (a) and hard mode (b) where x and x' are governed by the micropillar dimension.

For loading direction $\Phi_L = 90^\circ$ the analysis is complicated by the fact that both longitudinal and transverse systems operate simultaneously. According to the analysis in Figure 4.12, activity in transverse modes is found in isolated lamellae at even larger τ_{CRSS} of 620 MPa (see Table 4.1) and it hardly transmits to adjacent ones, suggesting that when both the plane and slip direction are oblique to the lamellar interfaces, slip transfer is not as easy as for mixed systems. This might be the reason for the higher strain hardening observed for $\Phi_L = 90^\circ$ (Figure 4.11), as opposed to the other deformation modes, $\Phi_L = 45^\circ$ (Figure 4.5) and $\Phi_L = 0^\circ$ (Figure 4.9). If this is the case, it is surprising to encounter pillar size effects for sizes well above the average lamellar thickness. However, the simultaneous activation of longitudinal slip, despite the low m for this deformation mode, might be the reason for the pillar size effects encountered for $D < 5 \mu\text{m}$. Indeed, if the τ_{CRSS} for longitudinal slip is determined from the yield stress computed for this loading direction, with a m value of 0.15, the variation of the τ_{CRSS} with pillar size (Figure 4.16) is identical to that obtained for a loading direction of $\Phi_L = 45^\circ$ (Figure 4.14), where only single longitudinal activity was observed. This supports the thesis that longitudinal activity dominates yielding for hard orientations close to $\Phi_L = 90^\circ$.

Fig. 4.16 Size effects for MPs with lamellar orientations close to 90° . τ_{CRSS} obtained through traces analysis, corresponding to longitudinal systems.

4.4 Conclusions

The work carried out in this chapter revealed a large plastic anisotropy at the microscale, comparable to that found in polysynthetically twinned crystals of other TiAl alloys at the macroscale.

The inspection of traces in the deformed specimens allowed to identify the type of slip/twinning activity operative at different lamellar orientations. For soft loading modes, with lamellar orientations close to $\Phi_L = 45^\circ$ with respect to the loading axis, the predominant slip/twinning system activated during deformation was of longitudinal type, that presents its plane of slip and Burgers vector parallel to the lamellar interfaces. The measured τ_{CRSS} necessary to promote the activation of longitudinal systems was relatively low, of the order of 209 ± 68 MPa and suffered negligible strain hardening, probably as a result of the lack of interaction with the lamellar interfaces. SEM and TEM confirmed the simultaneous activation of longitudinal slip and twinning systems at room temperature. In contrast, hard loading modes, for which $\Phi_L = 0^\circ$ and $\Phi_L = 90^\circ$, presented higher yield values. For the particular case where the lamellar orientation is close to $\Phi_L = 0^\circ$, the majority of plastic deformation was accomplished by mixed slip systems, whose slip planes intersect the lamellar

interfaces, but their slip direction remains contained within a lamella. The τ_{CRSS} needed for the activation of mixed deformation modes was higher than those measured for soft modes, of the order of 458 ± 9 MPa and suffered also little strain hardening, which together with the fact that slip traces were continuous but changed direction across several lamellae, seem to indicate that the lamellar interfaces are relatively transparent to mixed deformation modes. Micropillars with $\Phi_L = 90^\circ$ presented the hardest mode of deformation, associated with the activation of transversal systems, for which both the slip plane and slip direction are oblique to the lamellar interfaces. This loading mode presented higher strain hardening, that might derive from the high level of interaction between transverse systems and lamellar interfaces. The τ_{CRSS} for transverse deformation modes was determined to be 620 ± 33 MPa. However, it was not possible to isolate completely this deformation mode, because soft longitudinal systems were simultaneously activated for this loading direction, despite their almost negligible resolved shear stress. Furthermore, these results were used to calibrate a crystal plasticity subroutine that aims at capturing the mechanical response of isolated colonies as function of Φ_L , described in detail in Appendix A.

Finally, a study of micropillars size effects revealed that the results were insensitive to the pillar dimensions for pillar sizes above $5 \mu\text{m}$. The results are therefore representative of the behaviour that isolated colonies have on the overall behaviour of the polycrystalline specimens.

Chapter 5

Effect of lamellar refinement

This chapter aims at determining the influence of lamellar thickness on the strength of the fully lamellar Ti4522XD alloy under study. To achieve a lamellar refinement down to the nanometer scale regime, the starting centrifugally cast and HIPped alloy was subjected to water quenching, followed by a low temperature ageing treatment. The strengthening induced by the lamellar thickness reduction down to just a few nanometers, determined as a function of lamellar orientation by micropillar compression, was found to be very significant, even higher than what can be achieved by high Nb alloying.

5.1 Introduction

As described in Chapter 1, the most promising microstructure for high temperature structural applications is the so-called fully lamellar, formed by colonies of γ and α_2 lamellae. The development of FL microstructures requires heat treating at temperatures above the α -transus. The colony size (λ_C), the volume fraction of α_2 and γ phases, the lamellae width (λ_L), as well as the distance between domain boundaries (λ_D), all depend on the specific heat-treatment temperature and cooling rate. In particular, it is known that cooling rates below about 10°C s^{-1} favour the formation of lamellar microstructures whereas those above 100°C s^{-1} promote the stabilisation of the α or the α_2 phases at room temperature.

The average lamellae width is expected to play a key role in the yield strength (Lebensohn et al.). This strengthening effect resulting from internal boundaries, OV, PT and TT for lamellar interfaces and OV for domain boundaries, is often described in terms of a Hall-Petch

type relationship:

$$\sigma_y = \sigma_0 + k_y D^{-n} \quad (5.1)$$

where σ_y represents the yield stress, k_y is the Hall-Petch slope, and D a characteristic length that, depending on the loading mode, can be assigned to λ_L if $\Phi_L = 0^\circ$ or $\Phi_L = 90^\circ$ (hard loading modes) or to λ_D if $\Phi_L = 45^\circ$ (soft mode).

Various studies have been conducted with the aim of refining the lamellar spacing of γ -TiAl alloys down to the nanometer scale through heat treatments with the aim of enhancing their mechanical behaviour (Cao et al., 2000; Cha et al., 2008; Chan and Kim, 1995; Dimiduk et al.; Liu et al., 1996; Maruyama et al., 1997; Mine et al.; Qiang et al.; Sun, 1998; Wen et al.). Unfortunately, in the majority of the published work concerning the yield stress as a function of the lamellar spacing, the range of lamellar spacing was not sufficient to allow an unambiguous determination of σ_0 . To our knowledge, no previous works have focused on analysing the effect of average lamellar width λ on the orientation dependent strength of FL γ -TiAl alloys. This is precisely the objective of this chapter: to determine the anisotropy in the yield strength of FL γ -TiAl alloys as a function of the lamellar orientation Φ_L , for lamellae thickness in the nanoscale regime.

5.2 Experimental

In order to obtain a FL γ -TiAl with a lamellar thickness in the nanoscale regime, the as-received cast and HIPped material (see Section 3.1) was subjected to subsequent treatments. In particular, this alloy was heated at 10°C s^{-1} up to a temperature of 1390°C , kept at that temperature during 20 s, and finally water quenched at a measured cooling rate of $2000^\circ\text{C s}^{-1}$. This process, termed hereafter AWQ, was carried out in a Gleeble-3800 thermo-mechanical simulator (Figure 5.1), which allows to precisely control the temperature of the samples through a close-loop feedback control system via a fixed thermocouple of type R welded to the surfaces of the specimens, which are heated by the Joule effect. The AWQ specimen was then subjected to an ageing process aiming at strengthening the alloy through the precipitation of very refined lamellae. This was carried out at 500°C during 24 h on samples encapsulated in argon in order to avoid oxidation. Vickers hardness before and after the AWQ and ageing process was determined using a Shimadzu microhardness tester (Figure 5.2) at a load of 2 kg. This load was sufficient to produce a hardness impression that spans several colonies, so that to obtain an average response of each microstructure.

Fig. 5.1 Gleeble-3800 thermo-mechanical simulator system.

The microstructure and texture of the cast and HIPped alloy, as well as of the AWQ specimens, before and after the ageing treatment, were characterised, first, using a FEI Helios NanoLab 600i FEG-SEM (described in detail in Section 3.3.1) equipped with a NordlysNano EBSD detector. EBSD mapping was carried out at 20 kV and 4x4 binning with no frame averaging. Pseudo-symmetric misindexing in the analysis of $\gamma(\text{TiAl})$ was addressed through high accuracy band detection, as indicated in Section 3.3.4. More detailed microstructural characterisation, including local quantification of the chemical composition, was conducted in the FEG STEM FEI Talos F200. CBED and selected area electron diffraction (SAED) were applied for sample orientation and phase identification. Additionally, a nanoprobe was used for energy dispersive X-ray spectrometry (EDX) in high angle annular dark field (HAADF) STEM mode. TEM specimens were prepared by focused ion beam from colonies with zone axes of interest, which were previously identified by EBSD. X-ray diffraction (XRD) was also carried out in order to determine the lattice parameters and the weight volume fraction of the phases present. XRD was performed in a PANalytical X'Pert PRO diffractometer using $\text{CuK}\alpha$ radiation at 45 kV and 40 mA in the Bragg-Brentano geometry. A step size of 0.02° and a scan time of 2 s were used. Rietveld refinement (FullProf Suite software) using a pseudo-Voigt profile was performed on the XRD patterns. The Rietveld refinement fit was evaluated based on the agreement indices R_p and R_{wp} , as well as the goodness of the χ^2 fit.

Compression testing on micron-sized micropillars, milled out from selected isolated colonies of the aged specimen, was performed following the procedure described in Chapter 4 with the aim of analysing the effect of lamellar refinement and lamellar orientation on the

Fig. 5.2 Shimadzu microhardness tester.

mechanical behaviour. The MPs were milled with a square cross section, and dimensions of $5 \times 5 \times 10 \mu\text{m}^3$, in order to avoid buckling and size effects. Uniaxial compression testing of the MPs was carried out at room temperature using the TI 950 TriboindenterTM (Hysitron, INC., Minneapolis, MN). A flat-punch diamond tip with a diameter of $10 \mu\text{m}$ was installed on a high-load transducer to apply the force. The tests were carried out in displacement control mode at a programmed strain rate of 10^{-3} s^{-1} up to a maximum engineering strain of 10 %.

Fig. 5.3 PANalytical X'Pert PRO diffractometer.

5.3 Results and discussion

5.3.1 Microstructural characterisation

5.3.1.1 Cast and HIPped alloy

The characteristic fully lamellar microstructure of the cast and HIPped alloy is illustrated in Figure 5.4. In particular, Figure 5.4a is an orientation imaging map showing the intersection between two lamellar colonies. The alternating sequence of γ and α_2 platelets, being the latter present in a much smaller proportion, can be seen in the EBSD phase contrast map of Figure 5.4b. Additionally, Figure 5.4c shows the BC map. The measured colony size was $194 \pm 121 \mu\text{m}$, with values ranging from 52 to $636 \mu\text{m}$. A TEM foil was extracted from the cast and HIPped specimen, and oriented with a $\langle 1\bar{1}0 \rangle_\gamma \parallel \langle 11\bar{2}0 \rangle_{\alpha_2}$ zone axis in edge-on condition to minimise overlapping of lamellae. The corresponding SAED pattern is illustrated in Figure 5.4d, which is reproduced and indexed in Figure 5.4e,f in order to facilitate its interpretation. Figure 5.4e, in particular, depicts the spots corresponding to two twinned γ variants (dark and light grey spots) with a common $(111)_\gamma$ interfacial plane; Figure 5.4f illustrates the SAED pattern corresponding to α_2 lamellae, where $P6_3/mmc(194)$ space group absences, associated with 0001_{α_2} type reflections, are indicated in hollow circles. Comparison of Figure 5.4d and Figure 5.4f reveals that these absences were indeed experimentally observed, probably due to double diffraction given by the scattering of strong $\bar{1}100_{\alpha_2}$ reflections by $1\bar{1}01_{\alpha_2}$ planes. A more detailed examination of individual lamellae was carried out by TEM. Identification of individual γ and α_2 lamellae was conducted by selecting a $1\bar{1}00_{\alpha_2}$ type reflection, in DF mode (Figure 5.4g). EDX mapping of Ti and Al in the same area (Figure 5.4h and Figure 5.4i, respectively) reveals a Ti/Al proportion of 47.72/48.20 at.% for γ and α_2 phases, respectively (Table 5.1), as expected for stable $\gamma(\text{TiAl})$ and $\alpha_2(\text{Ti}_3\text{Al})$ compositions associated with low cooling rates, and in agreement with the TiAl binary phase diagram (see Figure 2.2). The thickness distributions associated with α_2 and γ lamellae, measured by TEM, were considerably broad, with average values $\lambda_{\alpha_2} = 319 \pm 39 \text{ nm}$ and $\lambda_\gamma = 287 \pm 133 \text{ nm}$.

Fig. 5.4 Microstructure of the cast and HIPped alloy: (a) EBSD Euler angle colouring map; (b) EBSD phase contrast map where γ and α_2 lamellae are indicated in red and blue, respectively; (c) Band contrast map; (d) Overlapping TEM SADPs of α_2 and γ variants coexisting within a single colony with shared zone axes $\langle 1\bar{1}0 \rangle_\gamma \parallel \langle 11\bar{2}0 \rangle_{\alpha_2}$; (e) Representation of the γ spots of the SADP of (d); (f) Representation of the α_2 spots of the SADP of (d); (g) DF image with highlighted α_2 lamella; (h) Ti composition map corresponding to the area shown in (g); (i) Al composition map corresponding to the area shown in (g).

Table 5.1 Chemical composition of the specimens under study measured by EDX in the TEM.

Specimen (Phase)	Ti (at.%)	Al (at.%)	Mn (at.%)	Nb (at.%)
Cast and HIPped (γ)	47.72	48.20	2.08	2.01
Cast and HIPped (α_2)	55.60	39.83	2.57	1.99
AWQ (α_2)	49.50	45.45	2.56	1.94
Aged (γ)	48.52	46.64	2.15	1.82
Aged (α_2)	55.85	39.59	3.39	1.84

5.3.1.2 Annealed and water quenched specimen

The AWQ specimen exhibits a microstructure formed by grains with irregular shapes, as shown in the EBSD orientation imaging map of Figure 5.5a. The average grain size was $6.90 \pm 3.12 \mu\text{m}$. The high cooling rate employed resulted in the stabilisation of a single α/α_2 phase due to the inhibition of γ nucleation. In order to determine the precise nature of the stabilised single phase, a TEM foil with a $\langle 1\bar{1}00 \rangle_{\alpha_2}$ zone axis was extracted from the AWQ sample. The corresponding SAED diagram is shown in Figure 5.5b. The differences in the intensities corresponding to $11\bar{2}0_{\alpha_2}$ and $22\bar{4}0_{\alpha_2}$ reflections are consistent with the long range ordering of Ti and Al atoms in the D0_{19} structure, thus confirming the presence of the α_2 phase. Additionally, the measured $(11\bar{2}0)_{\alpha_2}$ and $(0002)_{\alpha_2}$ interplanar distances ($d_{11\bar{2}0} = 2.87 \text{\AA}$ and $d_{0002} = 2.31 \text{\AA}$) are in agreement with those reported for the α_2 phase. TEM EDX mapping revealed a Ti/Al proportion of 49.50/45.45 at.% (Table 5.1), indicating supersaturation of Al in the α_2 intermetallic phase. Table 5.2 compares the lattice parameters of the α_2 phase in the cast and HIPped and AWQ specimens. It can be seen that the AWQ α_2 phase has a smaller unit cell volume.

Table 5.2 Results from the Rietveld refinement. Note that the goodness of the fitting might be limited by preference orientations in the bulk samples, as well as the background fitting.

Specimen (Phase)	a ($\text{\AA}$)	b ($\text{\AA}$)	c ($\text{\AA}$)	wt.%	R_p	R_{wp}	χ^2
Cast and HIPped (γ)	4.00692	4.00692	4.05331	94.5	7.62	11.44	3.61
Cast and HIPped (α_2)	5.75111	5.75111	4.62127	5.5			
AWQ (α_2)	5.73481	5.73481	4.61119	100	6.76	13.28	2.39

Fig. 5.5 Microstructure of the AWQ specimen: (a) EBSD Euler angle colouring map; (b) SADP corresponding to a single grain with a $1\bar{1}00_{\alpha:2}$ zone axis.

5.3.1.3 Aged specimen

Ageing of the AWQ sample resulted in the formation of a FL microstructure. Figure 5.6a is a TEM micrograph illustrating the developed nanolamellae. The lamellar spacing, measured as the average of 50 lamellae oriented in edge-on condition, were $\lambda_{\alpha_2} = 8 \pm 7$ nm and $\lambda_{\gamma} = 12 \pm 9$ nm with values within a range of 2 to 35 nm and 3 to 38 nm, for α_2 and γ , respectively. The average lamella thicknesses are, thus, at least one order of magnitude smaller than the values corresponding to the cast and HIPped material. The HAADF STEM images of Figure 5.6b, and Figure 5.6c confirmed the presence of a very refined lamellar microstructure, with a Ti/Al proportion of 48.42/46.64 at.% and 55.95/38.03 at.% for γ and α_2 phases, respectively (Table 5.1). These ratios are very similar to those reported for the cast and HIPped alloy.

Fig. 5.6 Nanolamellar structure of the aged alloy: (a) HAADF STEM image with its associated SADP in edge on condition; (b,c) The corresponding Ti and Al composition maps, respectively.

5.3.2 Micromechanical characterisation of the aged specimen

To gain insights into the effect that lamellar refinement has on the mechanical response of isolated colonies micropillar compression tests were carried out at different lamellar orientations (Φ_L). Figure 5.7 illustrates the engineering stress-engineering strain curves corresponding to micropillars with Φ_L values of 0° , 37° , 57° and 63° , whose images following testing are depicted in Figure 5.8, respectively. The MP with $\Phi_L = 0^\circ$ (hard loading mode) exhibits a yield stress value of 1762 MPa and an ultimate stress of 2635 MPa. This specimen undergoes significant hardening during the first stages of deformation, followed by softening at strains larger than 0.06. The latter might result from the development of instabilities caused by lattice rotation at high strain values as observed in the HR-SEM micrograph in Figure 5.8c. Two of the micropillars were compressed in soft loading modes, with $\Phi_L = 37^\circ$ and $\Phi_L = 57^\circ$ (Figure 5.8a,b). The corresponding yield stress and ultimate stress values are, respectively, 1420 MPa and 1434 MPa, and 2019 MPa and 2645 MPa. It must be noted that strain hardening is much more pronounced in the MP with $\Phi_L = 57^\circ$. This can be rationalised as follows. Longitudinal activity is favoured when Φ_L is close to 45° and its incidence decreases as Φ_L becomes smaller or larger than this value. Indeed, for the MP with

Fig. 5.7 Engineering stress-engineering strain curves corresponding to MPs with different lamellae orientations.

Fig. 5.8 HR-SEM micrographs of the compressed MPs with lamellae oriented at (a) 37°, (b) 57°, (c) 0° and (d) 63° with respect to the loading axis.

a lamellar orientation of $\Phi_L = 57^\circ$ and its incidence decreases as Φ_L becomes smaller or larger than this value. Indeed, for the MP with a lamellar orientation of $\Phi_L = 57^\circ$, the Schmid factors (m) associated to transverse and mixed systems, with a much smaller dislocation free path lengths and which therefore give rise to pronounced hardening, are higher than those of the MP with $\Phi_L = 37^\circ$, leading to higher interaction between non-coplanar slip systems with high m values (see Figure 5.9). The MP with $\Phi_L = 63^\circ$ exhibited a yield stress value of 1693 MPa and an ultimate stress value of 2350 MPa. As can be observed in the corresponding stress-strain curve in Figure 5.7, an initial hardening stage is followed by pronounced softening at strains larger than 0.035. The latter may result from the onset of longitudinal activity, as evidenced by the presence of traces parallel to the lamellar interfaces, pointed by red arrows in Figure 5.8d. As discussed in Chapter 4, during deformation in MPs with lamellae that are quasi-perpendicular to the loading axis, longitudinal slip often accommodates part of the strain imposed by the flat punch. Due to the characteristic low critical resolved shear stress values associated to these systems they are prone to activation

Fig. 5.9 Schmid factors as function of the lamellar orientation, with a resolution of 1° . (a) Longitudinal slip systems, (b) mixed slip systems and (c) transverse slip systems. The degree of freedom corresponding to a rotation about the direction normal to the interfacial plane has been considered, and scanned with a step size of 3° . (d) Superposition of the maximum m values corresponding to longitudinal, mixed and transverse slip systems. The black dashed lines in (d) indicate the maximum m values corresponding to MPs with lamellae orientations of 37° and 57° .

even when they possess relatively low m values, as in the case of $\Phi_L = 63^\circ$ MPs. Additionally, a second softening stage is visible for strains larger than 0.065, which might be attributed to interfacial sliding, leading to the onset of fracture. The resolved shear stress associated to this mechanism along the sliding plane (τ) amounts to 1012 MPa. Indeed, evidence of fracture has been identified in Figure 5.8d and is indicated by the white arrows.

The yield stresses measured from the aged MPs are compared in Figure 5.10 with those corresponding to the cast and HIPped material (Chapter 4) and with three additional TiAl alloys with different compositions (Ti-49.3Al (Fujiwara et al., 1990), Ti-45Al-8Nb (Peng et al.) and Ti-48Al (Kim et al., 1999b)). Clearly the dramatic lamellar refinement resulting from ultrafast cooling and ageing gives rise to a significant increase in strength for all the lamellae orientations investigated. This leads to an overall dramatic increase in hardness, from $323 \pm 10 \text{ HV}_2$ to $538 \pm 4 \text{ HV}_2$ in the AWQ and aged samples.

Fig. 5.10 Comparison of the yield strength values as a function of Φ_L corresponding to the AWQ and aged Ti4522XD alloy (present Chapter), cast and HIPped Ti4522XD alloy (Chapter 4), Ti-49.3Al (Fujiwara et al., 1990), Ti-45Al-8Nb (Peng et al.) and Ti-48Al (Kim et al., 1999b) alloys.

5.4 Conclusions

Exploring the microstructures resulting from ultrafast cooling and annealing, and their associated properties, has gained increased importance with the advent of AM technologies, in which a laser source is utilised to consolidate metallic powders to form 3D components. The solidification rates involved in AM technologies range from 10^3 to 10^8 °C s⁻¹. Development of powders with different compositions to those currently in use, which amount only to a handful of alloys, is of paramount interest to expand the possibilities of these technologies. This study shows that physical simulation might be an effective way to explore the potential of different alloy families as feedstock for AM. This first exploratory phase suggests that the Ti4522XD alloy might be a good candidate for AM.

In summary, this chapter confirms that ultrafast cooling and ageing of a Ti4522XD results in the transformation of a metastable α_2 phase, supersaturated in Al, into a fully nanolamellar structure with extraordinary strengths, regardless of the orientation of the lamellae with respect to the loading axis.

Chapter 6

Slip transfer across γ -TiAl lamellae

This chapter aims at determining the mechanisms governing dislocation transmission across lamellar interfaces in a Ti4522XD. With this aim, a microtensile grip was designed and fabricated, allowing to test small-sized specimens in tension. The deformation mechanisms acting in a microtensile specimen of a Ti4522XD alloy were investigated through EBSD and TEM. The results revealed that domain boundaries (OV) and lamellar interfaces (OV, TT and PT) are relatively transparent to dislocation propagation. While OV variants do not require a change in the Burgers vector of the outgoing systems due to a perfect geometrical compatibility between the incoming and outgoing slip systems, TT and PT interfaces require a change in the direction of the outgoing dislocation, which is, to a large extent, governed by the maximum geometrical alignment between the incoming and outgoing system.

6.1 Introduction

As introduced in Chapter 2, the extraordinary mechanical properties associated to fully lamellar γ -TiAl alloys derive, to a large extent, from the structure and chemical environment at the lamellar interfaces, which determines how plastic strain is accommodated (Bunshah et al.; Fu and Yoo, c; Kelly et al.; Lehoczky; Was and Foecke). The vast majority of plastic deformation in lamellar TiAl structures is accomplished by γ/γ boundaries, while α_2/γ interfaces provide the highest barrier strength to dislocation transfer, since pyramidal slip

requires extremely high shear stresses (Appel and Wagner; Appel et al., a; Singh et al., a,b; Sriram et al.; Vasudevan et al.; Wiezorek et al.). As discussed in Chapter 2, three types of interfaces between adjacent γ lamellae are possible, which can be described by rotations of 60° , 120° and 180° around the normal direction to the $\{111\}_\gamma$ interfacial plane, which are termed pseudo-twin, PT (60°), order variant, OV (120°), and true twin, TT (180°). Due to the tetragonality of the γ (TiAl) phase structure and the arrangement of Ti and Al atoms in its lattice, γ/γ interfaces present different interfacial energies (Γ) with a $\Gamma(180^\circ):\Gamma(120^\circ):\Gamma(60^\circ)$ ratio ranging from 1:2:3 to 1:6:7 (Fu and Yoo, a,b,c; Fu et al.; Girshick and Vitek; Hug et al., 1989; Inui et al., a; Lu et al.; Panova and Farkas; Rao et al., 1994; Simmons et al., a,b; Stucke et al.; Wiezorek and Humphreys; Woodward et al.; Yoo and Fu, a,b). Therefore, TT interfaces are fully coherent boundaries with the lowest associated interfacial energy, followed by order variant (OV) and pseudo-twin (PT) interfaces, whose associated energies are raised due to $c:a$ incompatibilities leading to the formation of misfit dislocations and ledges between regions of good atomic registry. Only in very thin lamellae a long-range atomic order is kept, being the misfit strains taken up by extremely high elastic stresses, homogeneously distributed along the lamellae (Shoykhet et al.).

Even though from energetic considerations the vast majority of γ/γ boundaries present in fully lamellar TiAl should be of TT type, processing conditions could alter the fraction of each boundary type, and this might have consequences on how plastic strain is accommodated. For instance, efficient slip transmission pathways across different lamellae might prevent plastic strain concentrations and ultimately delay fracture, improving ductility of TiAl alloys. However, studies of slip transmission across γ/γ lamellar interfaces are scarce.

In this chapter, slip transmission across different types of γ lamellar interfaces is experimentally analysed. For this, a micrometer scale specimen of fully lamellar TiAl, comprising a number of γ variants and their boundaries (including domain boundaries and TT, OV and PT lamellar interfaces), was milled by FIB within an individual colony and tested *in situ* under tension in the SEM. The orientation of each lamella and the character of each boundary was initially determined by EBSD, which was also used to follow the local plastic strain after testing. Finally, TEM studies allowed identification of the operating slip systems in each lamella, as well as the slip transmission paths across different boundary types. The observed slip transmission paths were then discussed in the light of the applied stress and geometrical considerations based on existing theories of slip transmission across grain boundaries. The final objective is to elucidate if some types of γ/γ boundaries present more efficient pathways for slip transmission than others.

6.2 Theoretical background in slip transmission across interfaces

With the aim of shedding light into the mechanisms governing dislocation propagation across interfaces and in order to develop slip transmission criteria extensive experimental research has been conducted during the six past decades (Abuzaid et al.; Aust et al.; Bollmann; Clark et al.; Lee et al., a,b; Lim and Raj; Livingston and Chalmers; Luster and Morris; Marcinkowski and Tseng; Seal et al.; Shen et al., a,b). These works are mainly focused on bicrystal deformation experiments, and are based on the observation of strained specimens in the TEM during or after experiments (Kacher et al.; Shen et al., a,b), and the transfer of slip traces generated by indentation tests through grain boundaries (Britton et al.; Lawrence et al.; Pathak et al.; SOER et al.; Wo and Ngan).

In general, the transmission of dislocations through grain boundaries is a result of dislocations reactions (Bollmann; Lim and Raj), the type of dislocations involved (Lee et al., a,b) and microstructural characteristics such as grain boundary type (Abuzaid et al.; Clark et al.; Kacher et al.; Seal et al.) and phase composition (Britton et al.; SOER et al.; Wo and Ngan).

Figure 6.1 summarises the basic dislocation interaction and slip transfer mechanisms expected to occur at a boundary where (a) illustrates the direct transmission of a dislocation with the same Burgers vector across a transparent interface without strengthening, (b) represents the direct transmission of a dislocation across a boundary with a change in the Burgers vector of the outgoing dislocation, leaving a residual boundary dislocation, (c) corresponds to the indirect transmission of a dislocation, where the outgoing slip system presents a different Burgers vector, leaving a residual boundary dislocation and (d) depicts an impenetrable boundary, where transmission is inhibited, leading to stress accumulations, localised rotations and pile-up of dislocations.

Numerous criteria based on geometrical parameters, stress and energetic functions or a combination of these have been proposed based on modelling and experiments. The parameters that geometrically define the slip transfer across a boundary are represented in Figure 6.2, where κ is the angle between the slip directions, θ is the angle between the two slip planes intersections with the grain boundary, ψ is the angle between the slip plane normal directions, γ is the angle between the direction of incoming slip and the plane normal of the outgoing slip, and δ is between the direction of the outgoing slip and the plane normal of the incoming slip. n , d and l are respectively the slip planes normal, slip directions and

Fig. 6.2 Geometrical parameters describing slip transfer across a boundary. Extracted from (Mercier et al.).

It is known that in multilayered structures the geometrical compatibility between slip systems in adjacent lamellae can lead the dislocation propagation mechanism assuming that there are no energetic aspects inhibiting transmission. That is, an interface can present a geometrically efficient pathway for a dislocation to propagate across it, but if it is weak in shear, it will present a large barrier to dislocation transmission. On the other hand, if the interface is resistant to sliding, transmission can be favoured even for a not perfectly aligned pathway. Beyerlein et al. have proposed an analytical equation that helps in quantifying how efficient a pathway is for the transmission of a dislocation based on the angle between the intersection lines that each slip plane makes with the interface (θ), the minimum angle between the Burgers vectors of both slip systems (κ) and the Schmid factor of the outgoing system (m_{out}). This approach considers that the propagation of dislocations is led by the coupling of two factors: (i) the minimum angle between the Burgers vectors of the incoming and outgoing slip systems, and (ii) the maximum m among the available systems in the destination lamella. The first factor is captured by expression 6.1, that considers the effect of the relative orientation of the incoming and outgoing systems, being the transfer mechanism favourable when the value of χ is close to unity:

$$\chi = \cos\left(\frac{\pi}{2} \frac{\theta}{\theta_c}\right) \cos\left(\frac{\pi}{2} \frac{\kappa}{\kappa_c}\right) \quad (6.1)$$

where θ_c and κ_c are the limiting angles for θ and κ respectively. The factor accounting for the effect of the Schmid factor of the available systems in the destination lamella is considered through m_{out} , leading to the expression $\chi_{\sigma} = \chi \cdot m_{out}$.

This criterion developed for bimetal interfaces conformed by two structures with a specific misorientation presents high similarities with γ/γ variants, whose relative orientation is described by the Blackburn orientation relationship. Thus, we have used this approach in the present chapter to study the mechanism governing the propagation of dislocations across interfaces in a deformed fully lamellar γ -TiAl specimen.

6.3 Experimental

With the aim of understanding the dislocation-interface interactions in the Ti4522XD γ -TiAl alloy under study in this thesis, a methodology for *in situ* tensile testing of micron-sized specimens was developed. For this, a custom-made diamond grip (Figure 6.3) was designed and fabricated out of a 5 μm flat punch in a dual-beam SEM-FIB Helios NanoLab 600i, FEI,

described in Section 3.4. A miniaturised specimen for tensile testing was milled out from an isolated colony at the edge of the sample, as indicated in Figure 6.4. Prior to milling the microtensile specimen, the sample required manual polishing to obtain a sharp edge. This process consisted on grinding two perpendicular surfaces with increasingly finer grit sizes, down to 4000 grit. Subsequently, a chemical-mechanical polishing step was carried out with a solution of colloidal silica (OP-S) and hydrogen peroxide (30 vol.%) in both surfaces of interest. Once the specimen was polished, it was mounted on a 52° pre-tilted sample holder to enhance handling of the specimen, allowing to position the surfaces of interest conforming the sharp edge perpendicularly to the ion beam by rotating the microscope stage 180° . Once mounted, the milling steps consisted on: (i) milling of a membrane at the edge of the sample with dimensions of about $5\text{ }\mu\text{m}$ in thickness, $20\text{ }\mu\text{m}$ in width and $20\text{ }\mu\text{m}$ in length, with ion currents ranging between 47 nA to 2.5 nA (see Figure 6.4a); (ii) subsequently, medium ion currents ranging from 0.79 nA to 0.23 nA were used to define the geometry of the microtensile specimen according to the shape of the diamond grip by milling out material perpendicularly to the wall obtained in step (i), avoiding the formation of sharp edges (see Figure 6.4b-c); and (iii), lower currents ranging between 40 pA to 80 pA and acceleration voltages down to 2 kV were applied for surface cleaning to enhance EBSD resolution of the external side of the specimen. The specimen was tested in an *in situ* PI87 Hysitron nanoindenter whose functioning is described in Section 3.5, in displacement control mode and a strain rate of

Fig. 6.3 Custom-made tensile diamond grip.

10^{-3} s^{-1} in a SEM chamber, allowing a precise positioning of the specimen with respect to the tensile grip, to minimise misalignments with the loading axis. Furthermore, the disposition of the specimen at the edge of the sample allowed for further crystallographic analysis through EBSD with a NordlysNano detector at 20 kV, 1 x 1 binning, no frame averaging, and a step size of 60 nm by tilting the specimen 70° with respect to the electron beam after testing.

Fig. 6.4 (a) Milling of a thin wall. (b) Geometry of the microtensile specimen. (c) Cleaning of the outer surface for EBSD mapping. Figure courtesy of Lingwei Yang.

Additionally, a TEM foil was extracted from the deformed specimen after failure, and was observed in a FEG S/TEM microscope (Talos F2000X, FEI). Convergent beam (CBED) and selected area (SAED) electron diffraction were conducted for sample orientation and phase identification in edge-on conditions, i.e., selecting a zone axis of $\langle 1\bar{1}0 \rangle_\gamma \parallel \langle 11\bar{2}0 \rangle_{\alpha_2}$ type, to allow for high resolution (HRTEM) imaging, and weak-beam was utilised to thoroughly identify the ordinary and superdislocation activity.

6.4 Results

6.4.1 Initial microstructure

The microtensile specimen presented a measured lamellar orientation of $\Phi_L = 49.6^\circ$ with respect to the loading axis (soft mode) and dimensions of $4.5 \times 4.5 \times 27 \mu\text{m}^3$. To further characterise the undeformed specimen EBSD data were taken prior to testing. The Kikuchi patterns associated with the different variants present in the specimen are indicated in Figure

6.5a, with their associated fundamental sector solution plotted in antipodal stereographic projections in Figure 6.5b, and their symmetrically equivalent rotated solution with the $(111)_\gamma$ pole perpendicular to the interfaces depicted in Figure 6.5c. As explained in Appendix B, the latter rotation facilitates the classification of the operating slip systems in each γ variant into longitudinal, mixed and transversal systems with respect to the lamellar interfaces, as they all share the same Miller indices, irrespective of the orientation of each variant. The orientation contrast map is presented in Figure 6.5d. The gauge length of the microtensile specimen was comprised by 3 α_2 lamellae, with thicknesses of 3200, 240 and 1250 nm, respectively, and 11 distinct γ lamellae, belonging to six different orientation variants (γ_1 - γ_6). Since some lamellae are formed by more than one orientation variant (separated by domain boundaries), each domain was denoted as γ_i^j where j refers to the lamella number (numerated from bottom to up) and i corresponds to the orientation variant. Note that the lamella denoted γ_1^* , at the bottom of the gauge length, belonged to a different colony. The orientation contrast map is depicted again in Figure 6.6, together with the band contrast map and the boundary misorientation map, where PT, TT and OV interfaces are depicted in dark blue (60° misorientations), light blue (70° misorientation) and dark red (90° misorientation), respectively. Interestingly the relative proportion of TT, OV and PT interfaces are 3.7, 93.2 and 3.1 %, respectively, despite the fact that TT interfaces are expected to be more numerous due to energetic considerations. As reported before, all domain boundaries belong to the OV type and contrary to the lamellar interfaces, are not parallel to $\{111\}_\gamma$ planes. Finally, it is also interesting to note that a few TiB_2 particles are also present in the gauge length of the specimen, as denoted by the arrows in Figure 6.6a.

Finally, it is instructive to compute the m values in each γ variant under the applied load, and these are gathered in Table 6.1. As expected for a lamellar orientation $\Phi_L = 49.6^\circ$ with respect to the loading axis (soft mode), there is always a longitudinal slip system with a m value close to 0.5, of ordinary type for variants γ_2 and γ_3 and of superdislocation type for variants γ_4 , γ_5 and γ_6 . Even though the m value in at least one of the transversal slip systems in each variant is also close to 0.5, it was expected that activity in the transversal slip systems would be hindered for this loading orientation. This is because the τ_{CRSS} of the transversal slip systems is much larger than for the longitudinal ones (Palomares-García et al.), based on the micropillar compression experiments presented in Chapter 4. However, as will be shown below, this was not the case and profuse transversal activity was also found in most of the γ variants. Moreover, Table 6.1 indicates that mixed slip systems present the lowest m values in the variants conforming the gauge length of the microtensile specimen

Fig. 6.5 Kikuchi patterns (a) and pole figures of α_2 and γ variants coexisting in the specimen corresponding to the as-indexed orientation description in (b) and the symmetrical rotated solution with the $(111)_\gamma$ planes parallel to the interfaces in (c). The IPF-Y contrast map of the undeformed specimen is depicted in (e).

Fig. 6.6 (a) Band contrast map of the undeformed specimen, with black arrows indicating the presence of two TiB_2 particles with higher associated BC values. (b) IPF-Y map with labelled lamellae and γ and α_2 variants. (c) Phase contrast EBSD map with interfaces identification.

(γ_2 - γ_6). Finally, it is interesting to note that, due to the polarity of twinning, this mechanism might also be activated for all variants, except for γ_5 , for which $[001]_\gamma$ is oriented parallel to the tensile axis, resulting in negative m values in all twinning systems.

Table 6.1 Schmid factors associated to the slip and twinning systems of the γ phase. Longitudinal and transverse systems in the variants conforming the gauge length of the specimen (γ_2 - γ_6) with the highest m have been highlighted in bold.

Slip	System	γ_1	γ_2	γ_3	γ_4	γ_5	γ_6
Longitudinal	(111)[$\bar{1}\bar{1}0$]	0.27	0.47	0.47	0.37	0.09	0.38
	(111)[$0\bar{1}1$]	0.00	0.37	0.38	0.10	0.47	0.09
	(111)[$\bar{1}01$]	0.27	0.10	0.09	0.47	0.38	0.47
Mixed	($\bar{1}\bar{1}1$)[$\bar{1}\bar{1}0$]	0.46	0.28	0.04	0.33	0.04	0.07
	($\bar{1}\bar{1}1$)[$0\bar{1}1$]	0.01	0.32	0.08	0.05	0.28	0.10
	($\bar{1}\bar{1}1$)[$\bar{1}01$]	0.45	0.05	0.10	0.28	0.33	0.02
Transverse	($\bar{1}\bar{1}1$)[110]	0.30	0.27	0.42	0.39	0.16	0.01
	($\bar{1}\bar{1}1$)[$\bar{1}10$]	0.43	0.49	0.08	0.32	0.11	0.33
	($\bar{1}\bar{1}1$)[011]	0.16	0.31	0.32	0.11	0.48	0.04
	($\bar{1}\bar{1}1$)[101]	0.41	0.16	0.16	0.27	0.39	0.43
	($\bar{1}\bar{1}1$)[$0\bar{1}1$]	0.16	0.39	0.02	0.16	0.26	0.13
	($\bar{1}\bar{1}1$)[$\bar{1}01$]	0.30	0.11	0.06	0.49	0.30	0.07
Twinning	System	γ_1	γ_2	γ_3	γ_4	γ_5	γ_6
Longitudinal	(111)[$11\bar{2}$]	0.16	0.16	-0.17	0.32	-0.49	-0.32
Transverse	($\bar{1}\bar{1}1$)[$1\bar{1}\bar{2}$]	0.35	0.21	-0.13	0.10	-0.47	-0.03
	($\bar{1}\bar{1}1$)[$\bar{1}\bar{1}\bar{2}$]	0.23	0.09	0.14	0.13	-0.39	-0.31
	($\bar{1}\bar{1}1$)[$\bar{1}\bar{1}\bar{2}$]	0.08	0.29	0.05	0.37	-0.32	-0.11

6.4.2 *In situ* microtensile tests

The engineering stress-displacement curves acquired during the microtensile tests are shown in Figure 6.7 (no attempt to compute strains was done due to machine compliance issues). The test was interrupted twice and the load removed at stress levels of 400, 650 and 897 MPa to acquire EBSD maps. Final fracture occurred during the 3rd loading step at 897 MPa. The corresponding SEM micrographs between tests are included in Figure 6.8a-e, where black arrows in Figure 6.8e indicate slip traces parallel to the lamellar interfaces, confirming the expected activation of longitudinal slip systems. The arrow in Figure 6.8d indicates the location of the final fracture, which took place very close to the thinnest α_2 lamella present in the gauge length, as will be shown in the TEM studies below.

Fig. 6.7 Stress-strain curves corresponding to the 1st, 2nd and 3rd tensile experiments.

It is interesting to note that the stress-strain curves obtained for 45° loading under tension differ substantially from those obtained under compression and shown in Figure 4.5. While under compression no strain hardening was substantial after initial yielding at 650 – 700 MPa, the level of strain hardening was relevant under tension from an initial yield stress of approximately 400 MPa up to a fracture stress of 900 MPa. Based on the

Fig. 6.8 SEM micrographs of the tensile specimen in the undeformed configuration (a), and deformed configurations (b-e). Fracture of the specimen is indicated with a black arrow in (d) and longitudinal traces revealed at the interfaces are indicated with black arrows in (e).

morphology of the deformed specimens, it is speculated that part of this difference could be attributed to the different constraints conditions encountered under tension and compression, as a result of the experimental set-up. Under compression, the activation of longitudinal slip resulted on a substantial lateral displacement of the head of the pillar (Palomares-García et al.), as shown in Figure 4.6, suggesting that sliding of the head of the pillar against the flat punch was possible. However, under tension, Figure 6.8 shows that the tensile grip suppresses any lateral displacement of the head of the specimen during testing, introducing a lateral constraint that might be responsible for the large strain hardening rate observed. It is not clear whether the constraint was due to the lateral stiffness of the experimental set-up or an uneven contact of the grip with the specimen head, but, contrary to the compression case, it resulted on the activation of the transversal slip systems, despite their large τ_{CRSS} (Palomares-García et al.), as will be shown in the TEM studies below. It is interesting to note that the specimen head rotated slightly counterclockwise (see Figure 6.8), which cannot be explained by the activation of longitudinal slip only.

6.4.3 Plastic strain localisation

The EBSD maps acquired after loading steps 1 and 2 did not reveal any major activation of twinning systems (see Figure 6.9a), which could result in substantial orientation changes of complete lamellae or large interface boundary migrations. In order to ascertain whether plastic deformation was mainly localised in some particular γ variants, Kernel average misorientation (KAM) maps were computed and are depicted in Figure 6.10. White arrows in the figure point at areas of high density of geometrically necessary dislocations, implying localisation of plastic strain. The areas with highest values of KAM are related to the two TiB_2 particles and the thinnest α_2 lamellae present in the gauge length (see Figure 6.6), where fracture of the specimen finally took place. It is clear from these maps that the α_2/γ interfaces and the TiB_2 particles represent strong barriers to dislocation transmission, which can trigger plastic strain accumulation, and eventually fracture. Interestingly, neither the γ lamellar interfaces (irrespective of their type) nor the domain boundaries seem to contribute to high values of KAM, suggesting that the γ/γ interfaces facilitate slip transmission.

Finally, band contrast (BC) maps before and after deformation are depicted in Figure 6.9b. It is interesting to note that, as pointed by the black arrows, some of the lamellae showed white bands that are coincident with transversal slip traces, conforming that transversal slip activity was taking place. As discussed above, the activation of transversal slip, that in

a) IPF-Y maps

b) BC maps

Fig. 6.9 (a) IPF-Y and (b) BC maps of the undeformed and deformed specimen, as indicated in the figure. Arrows in (b) point at transverse activity.

Fig. 6.10 KAM maps before and after testing with white arrows indicating where plastic deformation is concentrated.

principle should not be operative due to its larger τ_{CRSS} with respect to longitudinal slip, might be a result of the lateral constraint imposed by the experimental set-up.

6.4.4 Analysis of slip transmission at lamellar interfaces

The fact that the EBSD results suggested profuse longitudinal and transversal slip activity in the different γ variants, and that the γ/γ interfaces were not acting as impenetrable barriers to dislocations made this experiment very valuable to study slip transmission at γ/γ lamellar interfaces. Under the imposed loading conditions, longitudinal dislocations should interact with domain boundaries, of order variant (OV) type, while transverse dislocations could encounter OV, PT and TT lamellar interfaces in their paths. Unfortunately, the SEM, BC and KAM images depicted in Figures 6.8, 6.9 and 6.10 were not conclusive in determining transmission mechanisms at γ/γ boundaries, thus, requiring higher resolution in the inspection of the specimens. For this, a TEM foil was extracted parallel to the XY plane

of the specimen, and observed in edge-on conditions selecting a zone axis of $\langle \bar{1}\bar{1}0 \rangle_\gamma$ type with the aim of further understanding the dislocation activity and its interaction with the lamellar interfaces. Subsequently, weak-beam was utilised to thoroughly identify the ordinary and superdislocation activity. The most relevant observations are summarised in Figure 6.11.

The BF TEM micrograph depicted in Figure 6.11a shows the XY section of the deformed specimen at low magnification. This image accounts for the presence of dislocation activity parallel and transverse to the interfaces, and the presence of a TiB_2 particle that promotes a dislocation density higher than the average in its surroundings (Figure 6.11b), thus, being responsible for the low-quality indexing and high KAM values observed for this area in Figure 6.10, and confirming the role of TiB_2 particles as strong barriers for dislocation transmission that can trigger final failure. The letters in Figure 6.11a indicate the areas of the specimen where the rest of the higher magnification images were obtained from.

For instance, Figure 6.11c, obtained at the upper part of the specimen, accounts for the presence of two lamellae separated by an OV interface (γ_2^{10} and γ_5^{11}) that show profuse slip activity. γ^{10} is formed by the aggregate of three different domains comprising the following variants: γ_4^{10} , γ_5^{10} and γ_2^{10} , which are separated, as expected, by domain boundaries of OV type (compare with EBSD map of Figure 6.5). Dashed lines are a guide to the eye indicating slip traces that correspond to both longitudinal and transversal slip systems, as expected from the Schmid factor analysis of Table 6.1. Direct longitudinal slip transmission is clearly visible across domain boundaries. Regarding the transversal slip traces, those are highlighted with white arrows, and are labelled as 1 for lamella γ_2^{10} and 2 for lamella γ_5^{11} . The slip traces are perfectly aligned at both sides of the OV variant interface between γ_2^{10} and γ_5^{11} . SADPs confirmed that the observed traces correspond to $(\bar{1}11)[110]_\gamma$ for γ_2^{10} and $(1\bar{1}1)[011]_\gamma$ for γ_5^{11} . According to Table 6.1, these systems correspond to the maximum applied m values, with 0.49 and 0.48, respectively. No twinning activity was observed. This is not surprising for γ_5^{11} , for which, according to Table 6.1, twinning cannot be triggered by the applied stresses, but it does for γ_2^{10} . Since twinning has been observed in bulk TiAl samples, it is likely that the lack of twinning might be related to the small thickness of the γ lamellae present in the microtensile specimen, together with the large fraction of lamellae that were not well oriented for twinning (variants γ_5 and γ_6).

Figure 6.11d depicts a true twin interface between variants γ_2^6 and γ_3^5 . Despite longitudinal slip systems present the highest m values in both lamellae with a value of 0.47 (see Table 6.1), only transverse slip traces are observed. The transversal slip system with the highest m value in γ_2^6 corresponds to $(\bar{1}11)[110]_\gamma$, and is in agreement with the traces observed in the

Fig. 6.11 (a) HAADF STEM image of the deformed tensile specimen oriented in edge-on condition (a-d). The presence of TiB_2 particles is indicated with a white arrow in (b). High magnification micrographs of the area surrounding fracture are depicted in (c-h). (c) Depicts dislocation activity confined within lamellae and (d) transfer of twinning across a PT boundary.

micrograph. In contrast, the traces observed in γ_3^5 do not correspond to the system with the highest m value, $(\bar{1}\bar{1}1)[110]_\gamma$, but are parallel to either $(\bar{1}11)_\gamma$ or $(\bar{1}\bar{1}1)_\gamma$, with smaller m . Interestingly, longitudinal traces in α_2^3 confirms the activation of basal slip systems in this hard phase.

Furthermore, the area corresponding to the region above the crack is depicted in Figure 6.11e. In this figure, longitudinal traces corresponding to $(111)[\bar{1}01]_\gamma$ in γ_4^3 and $(111)[\bar{1}\bar{1}0]_\gamma$ in γ_3^2 are visible, with m values of 0.47 in both cases. Transverse slip propagates across the PT interface conformed between lamellae γ_4^3 and γ_3^2 in Figure 6.11e. The transversal systems with the highest m in γ_4^3 is in agreement with the observed traces, and corresponds to $(\bar{1}\bar{1}1)[101]_\gamma$. In contrast, the traces depicted in γ_3^2 correspond to a non-Schmid system parallel either to $(\bar{1}11)_\gamma$ or $(\bar{1}\bar{1}1)_\gamma$.

To further analyse the ordinary and super-dislocation activity, weak beam TEM was carried out in γ_2^1 lamella, in an area close to the crack, whose BF micrograph is depicted in Figure 6.12a, oriented with a $[001]_\gamma$ zone axis. This image reveals the highest concentration of dislocations close to the γ/α_2 interface, in agreement with the expected strong barrier character of this interface. Weak beam TEM was carried out in γ_2^1 lamella in an area close to the crack, whose BF micrograph is depicted in Figure 6.12a, oriented with a $[001]_\gamma$ zone axis. This image reveals the highest concentration of dislocations underneath the crack initiated in α_2 , that might derive in high stress levels in the surrounding area. Weak beam images with $\mathbf{g} = 220$, $\mathbf{g} = 200$ and $\mathbf{g} = 020$ are depicted in Figure 6.12b-d, respectively, corresponding to the extinction conditions for dislocation lines with Burgers vectors of $[\bar{1}\bar{1}0]_\gamma$ in Figure 6.12b, $[0\bar{1}1]_\gamma$ in Figure 6.12c, and $[\bar{1}01]_\gamma$ in Figure 6.12d. The micrograph in Figure 6.12b shows dislocation lines corresponding to transverse slip (white arrows) and dislocation debris (red arrows) that might contribute to strain hardening. Additionally, the longitudinal activity observed in Figure 6.12c and Figure 6.12d (black arrows) is extinct in Figure 6.12b ($\mathbf{g} = 220$). This confirms that longitudinal activity operating in γ_2^1 corresponds to ordinary dislocations with a Burgers vector $[\bar{1}\bar{1}0]_\gamma$ presenting the highest m among the longitudinal systems. In contrast, the fact that transverse activity does not become extinct in Figure 6.12b-d indicates that the observed transverse slip corresponds to both ordinary and super-dislocation activity.

Fig. 6.12 (a) Bright field (BF) micrograph of the area where fracture occurred, oriented with in a $[001]_\gamma$ zone axis. A high density of dislocations seems to accumulate at the interface between γ_2^1 and α_2^2 lamellae. Weak-beam images with $\mathbf{g} = 220$, $\mathbf{g} = 200$ and $\mathbf{g} = 020$ corresponding to extinction conditions for dislocation lines with Burgers vectors of $[1\bar{1}0]_\gamma$, $[0\bar{1}1]_\gamma$, and $[\bar{1}01]_\gamma$, are depicted in (b), (c) and (d).

6.5 Discussion of slip transmission across γ/γ boundaries

Overall, the TEM observations suggest differences between slip transmission across OV, PT and TT interfaces. While the observed slip systems are perfectly aligned and correspond to those with the maximum m value in the case of OV boundaries, the observed slip systems across PT and TT are not perfectly aligned and do not necessarily correspond to the slip systems with the maximum m value under the applied stress. Besides the energetic barriers deriving from the planar faults and stress accumulated at the interfaces, the geometrical compatibility between slip systems should play a role in the dislocation transfer mechanism of multilayer composites (Beyerlein et al.). In this sense, only order variant are $\Sigma 1$ interfaces (the two lamellae present coincident site lattices), suggesting that dislocations might be prone to transmit across OV interfaces, which present a complete set of compatible systems that do not require a change in the Burgers vector direction. In contrast, PT and TT interfaces require a switch in the Burgers vector for a dislocation to be transmitted across them since the minimum angle between $\{111\}_{\gamma}$ slip planes is close to 39° , depending on the specific $c:a$ unit cell parameter ratio, which depends on Al composition. Besides the reasoning based on the coincident site lattice character of an interface, that helps in identifying whether transmission may be favoured or not, the analytical approach suggested by Beyerlein et al., based on the computation of the parameters χ and χ_σ introduced in Section 6.2, provides a quantitative approach to estimate the efficiency of a pathway for a dislocation to be transmitted across an interface. The idea is to use this framework to help understanding the effect that the geometrical compatibility and the orientation of variants with respect to the loading axis have on the transfer of dislocations across interfaces.

In order to use this approach to analyse the probability of slip transmission across OV, PT and TT interfaces, several assumptions were made. First, only transversal slip and twinning were considered, because the longitudinal systems are parallel to the interfaces, and therefore, are not subjected to transmission. The calculations were particularised for $\theta_c = \pi/2$ and $\kappa_c = \pi/2$ in expression 6.1 with the aim of identifying the slip system with the highest probability of accomplishing propagation from one domain to the next. It was also assumed that the incoming dislocations always glided first in the thickest lamella out of the two conforming the interface and thus, transmission was always analysed from the incoming thickest lamella, γ_{in} , to the thinnest outgoing one, γ_{out} . Finally, the operative system corresponding to γ_{in} was chosen to be the one with the highest m value. Table 6.2 summarises the outcome of the calculations for the different interfaces and γ domains

encountered in the gauge length of the specimen. The slip systems with the maximum χ and χ_σ are in bold font in each case.

Table 6.2 χ and χ_σ values calculated for the different OV, PT and TT interfaces. The notation follows γ_{in}/γ_{out} (variant type). Only transverse systems (slip and twinning) have been taken into account. The operative incoming systems are indicated for each interface, corresponding to that with the highest m . Those twinning systems presenting a dash mean that their activation is inhibited and thus propagation across them has been neglected.

	γ_2/γ_4 (OV)		γ_2/γ_5 (OV)		γ_4/γ_5 (OV)		γ_2/γ_3 (TT)		γ_4/γ_3 (PT)		γ_4/γ_6 (TT)	
Transverse slip	$(\bar{1}\bar{1}1)[110]_{in}$ χ	$(\bar{1}\bar{1}1)[110]_{in}$ χ_σ	$(\bar{1}\bar{1}1)[110]_{in}$ χ	$(\bar{1}\bar{1}1)[110]_{in}$ χ_σ	$(\bar{1}\bar{1}1)[101]_{in}$ χ	$(\bar{1}\bar{1}1)[101]_{in}$ χ_σ	$(\bar{1}\bar{1}1)[110]_{in}$ χ	$(\bar{1}\bar{1}1)[110]_{in}$ χ_σ	$(\bar{1}\bar{1}1)[101]_{in}$ χ	$(\bar{1}\bar{1}1)[101]_{in}$ χ_σ	$(\bar{1}\bar{1}1)[101]_{in}$ χ	$(\bar{1}\bar{1}1)[101]_{in}$ χ_σ
$(\bar{1}\bar{1}1)[110]_{out}$	0.24	0.10	0.51	0.08	0.51	0.08	0.18	0.08	0.17	0.07	0.41	0.01
$(\bar{1}\bar{1}1)[110]_{out}$	0.26	0.08	0.25	0.03	0.25	0.03	0.35	0.03	0.34	0.03	0.43	0.14
$(\bar{1}\bar{1}1)[011]_{out}$	0.24	0.03	1.00	0.48	1.00	0.48	0.44	0.14	0.43	0.14	0.40	0.01
$(\bar{1}\bar{1}1)[101]_{out}$	0.51	0.14	0.24	0.10	0.24	0.10	0.84	0.13	0.83	0.13	0.17	0.07
$(\bar{1}\bar{1}1)[011]_{out}$	0.51	0.08	0.52	0.14	0.51	0.13	0.41	0.01	0.42	0.01	0.81	0.11
$(\bar{1}\bar{1}1)[101]_{out}$	1.00	0.49	0.26	0.08	0.25	0.08	0.40	0.02	0.41	0.02	0.33	0.02
Transverse twinning	$(\bar{1}\bar{1}1)[\bar{1}\bar{1}\bar{2}]_{in}$ χ	$(\bar{1}\bar{1}1)[\bar{1}\bar{1}\bar{2}]_{in}$ χ_σ					$(\bar{1}\bar{1}1)[\bar{1}\bar{1}\bar{2}]_{in}$ χ	$(\bar{1}\bar{1}1)[\bar{1}\bar{1}\bar{2}]_{in}$ χ_σ	$(\bar{1}\bar{1}1)[\bar{1}\bar{1}\bar{2}]_{in}$ χ	$(\bar{1}\bar{1}1)[\bar{1}\bar{1}\bar{2}]_{in}$ χ_σ		
$(\bar{1}\bar{1}1)[\bar{1}\bar{1}\bar{2}]_{out}$	0.49	0.05	—	—	—	—	—	—	—	—	—	—
$(\bar{1}\bar{1}1)[\bar{1}\bar{1}\bar{2}]_{out}$	0.07	0.01	—	—	—	—	0.11	0.01	0.38	0.05	—	—
$(\bar{1}\bar{1}1)[\bar{1}\bar{1}\bar{2}]_{out}$	0.41	0.15	—	—	—	—	0.76	0.03	0.46	0.02	—	—

Several general conclusions can be extracted from Table 6.2. For instance, in the case of OV interfaces, all incoming slip systems have a fully compatible outgoing system that keeps the direction of the Burgers vector ($\chi = 1$) and is as well oriented as the incoming system with respect to the applied stress (maximum χ_σ), as expected from the coincident site lattice argument used before. This is confirmed in Table 6.2 where γ_2/γ_4 (OV), γ_2/γ_5 (OV) and γ_4/γ_5 (OV) present one of the potential outgoing systems with $\chi = 1$, and the highest χ_σ . Moreover, for incoming dislocations of OD type, a switch to an SD system in the outgoing variant is needed, while incoming SD dislocations do not necessarily need to switch to OD dislocations to cross the interface (see Figure 6.5b where the pole figures corresponding to OV misoriented lamellae illustrate the compatibility between OD/SD, SD/OD, and SD/SD; but never OD/OD). Therefore, transmission can potentially take place leaving negligible dislocation residue at the interfaces, considering that $b_s \approx 2 \cdot b_o$, provided that two OD transmit into one SD and vice versa.

For the case of PT and TT interfaces, adjacent slip systems present a minimum interplanar angle of 39° , meaning that incoming and outgoing dislocations are not geometrically compatible and that a change in the Burgers vector is needed for a dislocation to propagate across these interfaces. Thus, for the case under study, the outgoing system in γ_2/γ_3 (TT), γ_4/γ_3 (PT) and γ_4/γ_6 (TT) with the highest geometrical compatibility, χ , might not be well oriented with respect to the applied load, and therefore it might not display the highest χ_σ (see Table 6.2). Furthermore, regardless of which the outgoing slip system is (that with the maximum χ or maximum χ_σ), transmission necessarily implies leaving a dislocation residue at the interfaces, and therefore, should be less favourable than for OV interfaces.

In addition to slip transfer, the compatibility of twinning across interfaces has been included in Table 6.2 for those variants whose m value associated to twinning have positive values, leading to τ values that could lead to their activation. Results of χ in Table 6.2 suggest that the variant type with the highest probability of accommodating propagation of twinning across its interface regardless of the outgoing m value is that of TT type, with the highest χ value corresponding to $\chi = 0.76$, followed by OV with $\chi = 0.49$ and PT with $\chi = 0.46$.

Despite the fact that slip and twinning transfer might be geometrically favourable for OV, PT and TT interfaces, there are numerous facts governing the transfer mechanism, such as the potential coherency stresses present at the interfaces and the elastic interaction between incoming dislocations and interfacial misfit dislocations. In the end, their relative importance is difficult to assess since they interact with each other, resulting in a complex coupling. This might be one of the reasons why twinning was not observed in any domain in the current study.

In any case, how do the calculations compare with the actual experimental observations? The most relevant observations and the comparison with the geometrical criteria developed above are summarised in the following sections, with the aid of diagrams that illustrate the incoming and outgoing slip traces.

6.5.1 OV interfaces and domain boundaries

Numerous experimental observations confirm that OV interfaces are favourable to slip transmission, as those shown in Figures 6.13 and 6.14, for transmission of transversal activity between $\gamma_5^{11}/\gamma_2^{10}$ and γ_2^4/γ_4^3 . In all cases, the observed incoming and outgoing systems present $\chi = \chi_{max} = 1$ and $\chi_\sigma = \chi_{\sigma-max} \approx 0.5$. Furthermore, dislocation lines parallel to the lamellar interfaces can be observed in γ_2^{10} and γ_5^{11} with m values of 0.47 in both cases, and the same can be said for the transfer of longitudinal slip across domain boundaries. Domain boundaries are always of OV type and transmission of longitudinal slip is profusely seen, for instance, between domains $\gamma_2^{10}/\gamma_5^{10}$ in Figure 6.13 and domains γ_2^4/γ_5^4 in Figure 6.14, because there always exist two longitudinal slip systems with $\chi = \chi_{max} = 1$ and $\chi_\sigma = \chi_{\sigma-max} \approx 0.5$.

Fig. 6.13 (a) IPF-Y map of the tensile specimen; (b) TEM image of the area surrounded by the circle in (a); (c) diagram illustrating transmission of longitudinal activity at the domain boundary $\gamma_2^{10}/\gamma_5^{10}$; (d) diagram illustrating transmission of transversal activity at the OV interface $\gamma_5^{11}/\gamma_2^{10}$.

Fig. 6.14 (a) IPF-Y map of the tensile specimen; (b) TEM image of the area surrounded by the circle in (a); (c) diagram illustrating transmission of longitudinal activity at the domain boundary γ_2^4/γ_3^4 ; (d) diagram illustrating transmission of transversal activity at the OV interface γ_2^4/γ_3^4 .

6.5.2 TT interfaces

The best example of slip transmission across a TT interface was found between lamellae γ_2^6/γ_3^5 in Figure 6.15. The observed incoming slip trace in γ_2^6 agrees with the one with the maximum m value (Table 6.1): $(\bar{1}\bar{1}1)[110]_{\text{in}}$ of ordinary type. According to the maximum values of χ and χ_σ for γ_2^6/γ_3^5 (TT) in Table 6.2, the outgoing system compatible with the observed traces in γ_3^5 is $(\bar{1}\bar{1}1)[101]_{\text{out}}$ of superdislocation type, with $\chi = \chi_{\text{max}} = 0.84$ and $\chi_\sigma = 0.13$. This slip system presents a m value of 0.16 versus $m_{\text{max}} = 0.42$ for the ordinary $(\bar{1}\bar{1}1)[110]_{\text{out}}$ with $\chi_\sigma = 0.18$, suggesting that the most efficient pathway for dislocation transfer across a TT boundary might be led by the geometrical compatibility between the incoming and outgoing systems, and not only the applied stress. Moreover, the $(\bar{1}\bar{1}1)[011]_{\text{out}}$ system, of superdislocation type, with $\chi = 0.44$ and $\chi_\sigma = 0.14$, is not observed, which confirms the important role of the geometrical compatibility between the incoming and outgoing systems. Furthermore, it is interesting to note that the Burgers vector of the residual dislocation

left at the TT interface between the observed incoming and outgoing system (assuming two incoming ordinary dislocations and one outgoing superdislocation) would be virtually contained in the $\{111\}_\gamma$ TT interface (not completely due to the tetragonality of the crystal structure), which is another factor that can contribute to this slip pathway.

Fig. 6.15 (a) IPF-Y map of the tensile specimen; (b) TEM image of the area surrounded by the circle in (a); (c) diagram illustrating transmission of transversal activity at the TT interface γ_2^6/γ_3^5 .

6.5.3 PT interfaces

The slip transmission mechanism observed in the PT interfaces is very similar to the TT interfaces, between lamellae γ_4^3/γ_3^2 , as shown in Figure 6.16. The observed incoming trace in γ_4^3 agrees with the one with the maximum m value (Table 6.1): $(\bar{1}\bar{1}1)[101]_{in}$, of superdislocation type. According to the maximum values of χ and χ_σ for γ_4^3/γ_3^2 (PT) in Table 6.2, the outgoing system compatible with the observed traces in γ_3^2 is $(\bar{1}\bar{1}1)[101]_{out}$ of superdislocation type, with $\chi = \chi_{max} = 0.83$ and $\chi_\sigma = 0.13$. This slip system presents a m value of 0.16 versus $m_{max} = 0.42$ for the ordinary system $(1\bar{1}1)[110]_{out}$, with $\chi = 0.17$, suggesting that

the most efficient pathway for dislocation transfer across a PT boundary is again led by the geometrical compatibility between the incoming and outgoing systems, and not only by the applied stress. Again, the fact that the $(\bar{1}\bar{1}1)[011]_{out}$, with $\chi = 0.44$ and $\chi_\sigma = 0.14$ is not observed, reinforces the view that the geometrical compatibility is a critical factor. Finally, the Burgers vector of the residual dislocations left at the PT interface between the observed incoming and outgoing systems (assuming one incoming superdislocation and one outgoing superdislocation) would be contained in the $\{111\}_\gamma$ PT interface, which is another factor that can contribute to this slip pathway.

Fig. 6.16 (a) IPF-Y map of the tensile specimen; (b) TEM image of the area surrounded by the circle in (a); (c) diagram illustrating transmission of transversal activity at the PT interface γ_4^3/γ_3^2 .

6.6 Conclusions

Tensile testing of a micron-sized γ -TiAl specimen with a lamellar orientation of $\Phi_L = 49.6^\circ$ with respect to the loading axis (soft mode) provided useful insights on the interaction between dislocations and interfaces.

Firstly, it was observed that TiB_2 particles represent strong barriers to dislocation transmission, deriving in a higher density of geometrically necessary dislocations and localisation of plastic strain, that can ultimately lead to fracture, limiting the ductility of the alloy.

Moreover, the dislocation activity observed under tension in soft mode was almost exclusively slip of ordinary and superdislocations with no preference between them. There was no evidence of twinning, that could be inhibited due to the small thickness of the γ lamellae of the tested specimen. In general terms, slip transmission events have been extensively observed across the domain boundaries (OV) and lamellar interfaces (OV, TT and PT), which seem to be relatively transparent to dislocation transmission regardless of their type. Geometrical analyses confirmed that OV interfaces present fully compatible slip systems, that do not require a change in the outgoing Burgers vector. In contrast, TT and PT interfaces do require a switch between the incoming and outgoing Burgers vector since the geometrical compatibility is not perfect. Despite this, mechanisms governing the activation of the outgoing slip system seem to be determined by the highest geometrical alignment between the incoming and outgoing systems.

These observations might be relevant for the ductility of TiAl alloys, as from the viewpoint of slip compatibility, a larger fraction of OV interfaces should favor ductility over PT and TT interfaces.

Chapter 7

Conclusions

The work completed during the course of this thesis has broadened the existing knowledge on the mechanical behaviour of a Ti4522XD alloy at the colony level through (i) the compression of micropillars at different lamellar orientations (Φ_L), (ii) the refinement of lamellar thicknesses distribution and the study of its effect on the mechanical response of the alloy at the micro-scale level, and (iii) the study of the tensile behaviour of isolated colonies and dislocation-interface interactions. Furthermore, the latest approach involved the development of an *in situ* testing methodology by designing and fabricating a grip from a commercial diamond flat punch that can be applied for the study of micron-sized specimens under tension and tension/compression cyclic loads.

The main conclusions extracted from the previous work are summarised as follows:

- Compression of micropillars at room temperature revealed a large plastic anisotropy at the microscale, comparable to that found in polysynthetically twinned crystals of other TiAl alloys at the macroscale. Based on slip/twin trace analysis, the following conclusions were obtained:
 - Loading at $\Phi_L = 45^\circ$ with respect to the lamellar orientation constitutes a soft mode that resulted in the activation of longitudinal deformation systems, with both the slip plane and the slip direction are parallel to the lamellae. The τ_{CRSS} for the activation of longitudinal deformation modes was relatively low, of the order of 209 ± 68 MPa and suffered negligible strain hardening, probably as a result of the lack of interaction with the lamellar interfaces. Analysis of the

deformed pillars in the SEM and the TEM and of the strain bursts indicated that both longitudinal twinning and slip systems might be activated at room temperature.

- Loading at $\Phi_L = 0^\circ$ with respect to the lamellar orientation activates harder modes of deformation, mainly mixed deformation modes, either twinning or slip, by which shear is concentrated on planes that are oblique to the lamellar interfaces but on directions that remain parallel to them. The τ_{CRSS} for the activation of mixed deformation modes was higher, of the order of 458 ± 9 MPa and suffered also little strain hardening, which together with the fact that slip traces were continuous but changed direction across several lamellae, seem to indicate that the lamellar interfaces are relatively transparent to mixed deformation modes.
 - Loading at $\Phi_L = 90^\circ$ with respect to the lamellar orientation activates the hardest modes of deformation, associated with the activation of transversal deformation modes, for which both the slip plane and slip direction are oblique to the lamellar interfaces. Those systems seem to display the larger interaction with lamellar interfaces, since only isolated transversal twins could be found in individual lamellae, that did not propagate across lamellar interfaces. As a result, this deformation mode showed the largest strain hardening behaviour. The τ_{CRSS} for transverse deformation modes was determined to be 620 ± 33 MPa. However, it was not possible to isolate completely this deformation mode, because soft longitudinal systems were simultaneously activated for this loading direction, despite their almost negligible resolved shear stress.
 - A through study of pillar size effects revealed that the results were insensitive to the pillar dimensions for pillar sizes above $5 \mu\text{m}$.
 - The experimental results extracted from micropillar compression tests can be successfully applied for developing mesoscale plasticity models that capture the micromechanics of fully lamellar γ -TiAl microstructures at larger length scales.
- Lamellar refinement down to the nanometer scale regime was achieved by subjecting the starting centrifugally cast and HIPped alloy to water quenching, followed by a low temperature ageing treatment. Subsequently, micropillar compression tests were conducted to study the effect of lamellar refinement on the mechanical properties of isolated colonies, leading to the following conclusions:

-
- Ultrafast cooling and ageing of a Ti4522XD results in the transformation of a metastable α_2 phase, supersaturated in Al, into a fully nanolamellar structure.
 - The refinement of the lamellar structure down to the nanometer scale resulted in extraordinary strengths, regardless of the orientation of the lamellae with respect to the loading axis.
 - Physical simulation might be an effective way to explore the potential of different alloy families as feedstock for AM since the cooling rate achieved in this work is of the order of the solidification rates involved in AM technologies.
- The deformation of fully lamellar γ -TiAl in tension at the colony level was studied through the development of a device for *in situ* testing of microtensile specimens, revealing the role of the domain and lamellar boundaries on strain propagation:
 - α_2 lamellae and TiB_2 particles (added to Ti4522XD to limit the colony size during casting) represent strong obstacles to dislocation transmission, inducing lattice distortions in the vicinity of these phases, that can ultimately lead to fracture, limiting the ductility of the alloy.
 - The deformation mechanisms observed under tension at 45° with respect to the lamellar orientation, were almost exclusively slip of ordinary and superdislocations, with no twinning activity. Since twinning has been observed in bulk TiAl samples, it is likely that the lack of twinning might be related to the small thickness of the γ lamellae present in the microtensile specimen, together with the large fraction of lamellae that were not well oriented for twinning. No preference for ordinary or superdislocation activity was found, probably because the applied stresses are so high that morphological considerations (interaction between dislocation and lamellar interfaces) are more important than the character of the dislocations.
 - Despite the fact that γ/γ lamellar interfaces and domain boundaries act as barriers against dislocation slip (as evidenced by the strengthening observed with lamellar width reduction in Chapter 5), the slip compatibility between different orientation domains of the γ phase present within each single colony seem to be fairly good. As a matter of fact, slip transmission events have been extensively observed across domain boundaries as well as γ/γ interfaces of OV, PT and TT type. However, the slip transfer pathways differ substantially for each type of interface, and the observations have been rationalised based on geometrical considerations.

- In the case of OV boundaries (both domain boundaries and interfaces between γ/γ), slip transmission pathways are geometrically fully compatible, so that the incoming and outgoing slip systems present a perfect alignment and are well oriented with respect to the applied stress.
- In the case of TT and PT interfaces, the geometrical alignment between incoming and outgoing slip systems is not perfect, but plays an important role on determining which slip systems are activated. As such, the outgoing slip system observed in most cases is that with the maximum geometrical alignment, in favor of other slip systems that are better oriented with respect to the applied stress.
- The observations regarding dislocation-interface interactions might be relevant for the ductility of TiAl alloys, as from the viewpoint of slip compatibility, a larger fraction of OV interfaces should favor ductility over PT and TT interfaces.

Chapter 8

Future work

Due to the small thickness of the lamellae in γ -TiAl alloys, the large number of interfaces present in the microstructure can determine, to a large extent, the deformation mechanisms. Because of this, it is important to broaden the work conducted in the present thesis in order to better understand the behaviour of γ -TiAl alloys. For this, the following areas of future work are envisioned:

- Further work could be conducted in understanding the tensile behaviour of micron-sized specimens at different loading modes. In particular, it would be desirable to broaden the work conducted in Chapter 6 with specimens loaded at hard modes, i.e., $\Phi_L = 0^\circ$ and $\Phi_L = 90^\circ$.
- The working temperature for the projected applications of γ -TiAl alloys is close to 750°C . In order to complement the work of this thesis it is proposed to conduct a full characterisation of the deformation behaviour of the material at high temperature. This could be conducted through compression of micropillars at different lamellar orientations (Φ_L) with the aim of identifying the predominant deformation mechanisms at different loading modes. Previous work conducted confirmed that the micron-sized specimens suffer from oxidation at high temperatures, because of this, it is suggested to carry out these experiments in a vacuum environment.
- Once the micromechanical characterisation of the alloy is fully understood at temperatures at to 750°C it would be possible to analyse the pre-yielding phenomenon observed in the polycrystalline bulk alloy. This has been reported to be caused by

strain incompatibilities between adjacent colonies with different lamellar orientations with respect to the loading axis. In fact, the previous work conducted in this thesis towards the calibration of a crystal plasticity subroutine, that captures the deformation behaviour of isolated colonies at different lamellar angles (Φ_L), can serve as a starting point in the development of a mesoscale model in which the strain incompatibilities between colonies in polycrystalline specimens is studied.

Appendix A

Calibration of a crystal plasticity subroutine

A.1 Introduction

As discussed along the thesis, the micromechanical parameters determined in this work can be very useful to develop a microstructurally based model to predict the mechanical behaviour of fully lamellar TiAl alloys. This appendix presents preliminary work in this direction. With this aim, a standard crystal plasticity (CP) model (Segurado and Llorca) was used to simulate the plastic behaviour of the γ -phase variants of the alloy of interest, taking into account their FCT crystallographic structure and Blackburn orientation relationship (Table 2.3) between adjacent lamellae.

A.2 Brief description of the crystal plasticity model

The CP model relies on the multiplicative decomposition of the deformation gradient (Lee), $\mathbf{F}$, into the elastic, $\mathbf{F}^e$, and plastic part, $\mathbf{F}^p$, following the expression:

$$\mathbf{F} = \mathbf{F}^e \mathbf{F}^p \tag{A.1}$$

where $\mathbf{F}^p$ corresponds to the so-called relaxed configuration.

Taking into account the definition of velocity gradient, $\mathbf{L} \equiv \nabla_x \nu = \dot{\mathbf{F}}\mathbf{F}^{-1}$, expression A.1 leads to:

$$\mathbf{L} = \dot{\mathbf{F}}\mathbf{F}^{-1} = \mathbf{L}^e + \mathbf{F}^e \mathbf{L}^p \mathbf{F}^{e^{-1}} \quad (\text{A.2})$$

being $\mathbf{L}^e$ and $\mathbf{L}^p$ the elastic and plastic velocity gradients, respectively.

As the plastic deformation is defined by the glide on the different slip systems $\mathbf{L}^p$ is calculated by the sum of the shear rates, $\dot{\gamma}^\alpha$ of each slip system, α , as:

$$\mathbf{L}^p = \sum_{\alpha} \dot{\gamma}^\alpha (\mathbf{s}^\alpha \otimes \mathbf{m}^\alpha) \quad (\text{A.3})$$

where $\mathbf{s}^\alpha$ and $\mathbf{m}^\alpha$ stand for the unit vectors in the slip direction and perpendicular to the slip plane, respectively.

The plastic behaviour of the crystal, $\dot{\gamma}^\alpha$, is assumed to be that of an elasto-viscoplastic solid, which is described by the following power-law (Asaro and Needleman; Peirce et al.) expression:

$$\dot{\gamma}^\alpha = \dot{\gamma}_0 \left(\frac{|\tau^\alpha|}{g^\alpha} \right)^{\frac{1}{m}} \text{sgn}(\tau^\alpha) \quad (\text{A.4})$$

being $\dot{\gamma}_0$ and m the reference shear strain rate and the rate-sensitive exponent respectively (Hutchins on, 1976). The evolution of the critical shear stress, g^α , for a given slip system, α , can be obtained from the classical hardening model of Asaro and Needleman as follows:

$$g^\alpha = \sum_{\beta} q_{\alpha\beta} h(\Gamma) \dot{\gamma}_\beta \quad (\text{A.5})$$

where $q_{\alpha\beta}$ with $\alpha = \beta$ and $\alpha \neq \beta$ are the self- and latent-hardening coefficients, respectively, and h is referred to as hardening modulus:

$$h(\Gamma) = h_0 \text{sech} \left(\frac{h_0 \Gamma}{\tau_s - \tau_0} \right) \quad (\text{A.6})$$

where h_0 is the initial hardening modulus, τ_0 is the initial yield shear stress, τ_s the saturation yield shear stress, and Γ is the accumulated shear strain in all slip systems expressed by:

$$\Gamma = \int_0^t \sum_{\alpha} \dot{\gamma}^\alpha dt \quad (\text{A.7})$$

The resolved shear stress, τ^α , for a given slip system, α , is obtained as the projection of the Kirchoff stress on that system according to the following expression:

$$\tau^\alpha = \mathbb{C} \left[\left(\mathbf{F}^{eT} \mathbf{F}^e \right)^{1/2} - \mathbf{I} \right] : \mathbf{s}^\alpha \otimes \mathbf{m}^\alpha \quad (\text{A.8})$$

where $\mathbb{C}$ represents the fourth-order elastic stiffness tensor of the crystal and $\mathbf{I}$ the second-order identity tensor.

Finally, the Cauchy tensor, $\boldsymbol{\sigma}$, is obtained from the second Piola-Kirchoff tensor assuming small elastic deformations.

A.3 Constitutive material parameters

For the numerical simulations the effect of the α_2 -phase on the plastic deformation of the MPs was neglected, as it has been reported by other authors that deformation is mainly carried out by the softer γ -phase which is present in the material in a proportion of 80 – 95 vol.% regardless of whether the alloy has a lamellar or equiaxed microstructure (Appel and Wagner; Appel et al., a). In contrast, the α_2 -phase is thought to accomplish deformation by brittle fracture and does not seem to play a role in the deformation of the lamellar microstructure.

The elastic constants for $\gamma(\text{TiAl})$ were obtained from He et al., *i.e.*, $C_{11} = 183 \text{ GPa}$, $C_{33} = 177 \text{ GPa}$, $C_{12} = 75 \text{ GPa}$, $C_{23} = 75 \text{ GPa}$, $C_{44} = 104 \text{ GPa}$, $C_{66} = 77 \text{ GPa}$. The values of the self- and latent-hardening parameters, $q_{\alpha\beta}$, were defined according to the experimental evidence where multiple slip across mixed systems of the same m is observed, the estimated values are specified in Table A.1. The initial yield stress, τ_0 , the saturation yield stress, τ_s , the initial hardening modulus, h_0 , and the saturation hardening modulus, h_s , were calculated applying an iterative approach for each set of systems based on the recorded stress-strain curves from the MP compression tests, the final values are indicated in Table A.2 where the saturation hardening values represent the effect of the interaction between a given set of systems with the interfaces. Moreover, in order to match the simulations with the experimental set up a reference initial shear rate $\dot{\gamma}_0$ of 10^{-3} s^{-1} was chosen, with a rate-sensitive exponent m value of 5.

For the definition of the set of systems of the f.c.t. γ -phase it was assumed the morphology-based classification reported by Lebensohn et al. which is considered to play a key role in fully lamellar microstructures. Three sets of slip systems were defined, namely longitudinal slip systems (with slip directions and planes parallel to the lamellae interfaces), mixed slip

Table A.1 Estimated values for the cross-hardening parameters q_{ij} .

	longitudinal	mixed	transversal
longitudinal	1	1	1
mixed	1	2	1
transversal	1	1	1

Table A.2 Crystal plasticity model parameters for each slip system.

Slip system	τ_0 (MPa)	τ_s (MPa)	h_0 (MPa)	h_s (MPa)
longitudinal	209	254	14000	0
mixed	458	485	14000	0
transversal	620	668	5000	1000

systems (with slip directions parallel to the lamellae interfaces, and slip planes transverse to the interfaces) and transversal slip systems (with slip directions and planes transverse to the interfaces) as indicated in Table 2.4 and Table 2.5, where the $(111)_\gamma$ plane has been chosen to be parallel to the lamellae interfaces (Blackburn, 1970). By assuming this classification no distinction between ordinary and super-ordinary dislocations is considered. Also, twinning deformation systems were not taken into account in the numerical model presented in this work due to several considerations described as follows:

The main effect that mechanical twinning might have on the overall response of the MP can be classified in two main contributions, *i.e.*, re-slip and strengthening. This should be analysed for the cases where longitudinal and transversal activity is to be expected, as they are the only modes of deformation which share both slip and twinning systems. The contribution of re-slip and strengthening on the deformation behaviour of the MP will present different levels of implication depending on the lamellar orientation Φ_L . That is, for soft loading modes where longitudinal twinning might take place along $(111)_\gamma$ planes, the presence of re-slip is not expected to have any significant effect on the mechanical response of the system as true-twin boundaries are already present in the material, formed during precipitation of the γ -phase, thus there exists already adjacent lamellae with a true-twin misorientation. Also, twinning might lead to a reduction of the lamellae width but, as longitudinal systems present the plane and direction of slip/twinning parallel to the interfaces, the characteristic length which governs this mode of deformation will not be affected and thus strengthening is not likely to be observed. For the case of hard loading modes with lamellar angles Φ_L close to 90° transversal systems are likely to carry out yielding. If mechanical twinning took place

along lamellae the potential re-slipped volume fraction of the material would be limited due to constrictions imposed by the lamellar spacing so no relevant softening might occur. In addition, as transversal activity is mainly concentrated in certain lamellae for low strain values (up to 10%) strengthening might not happen as there will be enough undeformed lamellae able to assume deformation in an easier way (*e.g.*, through longitudinal slip as described in Section 4.3.1.3).

Also, mechanical twinning in γ -TiAl has been reported not to cause any significant difference in yield stress between tension and compression. This could be attributed both to the broad distribution of γ variants and the switching of the deformation mode from twinning to slip and vice versa in each γ domain due to the change in the sense of loading.

Furthermore, simultaneous activity between twinning and slip systems of the same mode has been observed in this work, where no evidence of isolated mechanisms either slip or twinning has been reported to take place (Inui et al., 1992b).

To conclude, the omission of twinning in the numerical model will not make any significant change in the results due to the following considerations that summarise the discussion presented above:

- (i) The effect of mechanical twinning in this particular multilayered structure presents a relatively low implication on the mechanical response of the MPs, neither re-slip nor strengthening are to be expected to modify the properties of the material substantially.
- (ii) The polar nature of mechanical twinning can be neglected in the numerical model as there are multiple compatible twinning/slip systems, which activate depending on the sense of loading.
- (iii) The τ_{CRSS} values of twin and slip systems belonging to the same type of deformation, i.e., longitudinal or transversal, can be considered to be comparable based on our work and previous results which show simultaneous activity of both twinning and slip.

A.4 Finite element model and boundary conditions

The constitutive crystal plasticity model described above with a time integration procedure based on the Newton-Raphson approach was implemented in the commercial FEM software ABAQUS using a user material subroutine (UMAT).

Three 3D models of square MPs were designed corresponding to lamellar orientations of 0, 45 and 90 degrees (see Figure A.1). Dimensions of the pillars were set to 5 μm width, 10 μm

height. No taper was considered in the CAE design as its influence in the numerical results (for values lower than 5° , as those experimentally observed) is smaller than the dispersion obtained from the experiments which might be attributed to variations of microstructural features between different MPs. The fillet radius of the base for all the models was chosen to be $1\text{ }\mu\text{m}$, which is similar to that measured from the milled pillars. The MPs were placed on a large substrate with the same crystal orientation and hardening parameters as one of the lamellae of the γ -phase variants present in the model, *i.e.*, Mat-1, Mat-2, Mat-3 or Mat-4; which represent the four possible crystallographic orientations established by the Blackburn relationship (Blackburn, 1970). This gives raise to three interface variants, *i.e.*, pseudo-twin (between Mat-1/Mat-2 and Mat-3/Mat-4), order-variant (between Mat-1/Mat-3 and Mat-2/Mat-4) and true-twin (between Mat-1/Mat-4 and Mat-2/Mat-3) (see Figure A.1). The material assignment described here only takes into account the effect of the local crystallographic orientation of each lamellae on the activation of the different slip systems due to the tetragonal distortion of the lattice, but the model does not take into account the properties of the material at the interfaces.

Fig. A.1 CAE models of three micropillars with lamellar orientations of 0° , 45° and 0° .

Table A.3 summarises the Euler angles and miller indices of the crystal lattices which define the four materials of the models. The values are given for a global cartesian coordinate system represented in Figure A.1 for each case, where the 1-axis is parallel to one of the horizontal edges of the pillars and thus, the 1-axis is always perpendicular to the $[111]_\gamma$ direction.

Table A.3 Orientation of the materials considered in the CAE models.

Material	φ_1	ϕ	φ_2	3-axis (hkl)	1-axis [uvw]
Mat-1	0°	55°	45°	(111) $_{\gamma}$	[$\bar{1}10$]
Mat-2	300°	55°	45°	(111) $_{\gamma}$	[10 $\bar{1}$]
Mat-3	120°	55°	45°	(111) $_{\gamma}$	[$\bar{1}01$]
Mat-4	180°	55°	45°	(111) $_{\gamma}$	[$\bar{1}10$]

Lamellae thicknesses were chosen to be about 1/10 of the total length of the pillar for all the CAE models. The relative disposition of the γ variants is shown in Figure A.1, where the colour code indicates the material assignment for each lamella.

The flat punch was treated as an analytical rigid body, and a coefficient of friction between the indenter and the top surface of the MP of 0.02 was chosen in order to reduce shape instability irrespective of the initial crystallographic orientation of the compressed specimen (Raabe et al.).

The mesh was refined within the pillar to ensure that reliable results were obtained following a mesh convergency study in order to minimise the computational requirements. In the final model 4 hexahedral elements in the transverse direction were chosen for each lamella. The mesh for each model consisted of 16000, 15600 and 16000 C3D8 lineal hexahedral elements for the pillars with lamellar angles Φ_L of 0°, 45° and 90°, respectively; and 14065, 15391 and 15013 C3D4 linear tetrahedral elements for the substrate in the first, second and third model respectively.

In the formulation of the boundary conditions the nodes at the base of the substrate were fixed and any displacement and/or rotation were restricted. The rigid body reference point was allowed to undergo a prescribed displacement of 1 μm in the axial direction of the MP. The total duration of the simulation was set to 100 s to match the parameters of the elasto-viscoplastic law with the experimental set up.

A.5 Results and discussion

The numerical simulation results for the MP model with $\Phi_L = 0^\circ$ are presented in a black dashed line in Figure A.2. This curve shows a good fitting with the experimental recorded curves. CPFEM modelling allowed us to predict the evolution of the different slip systems during the compression testes, and their instantaneous contribution to the total accumulated plastic shear after matching the constitutive model parameters. As can be seen in Figure

Fig. A.2 Experimental and CPFEM (dashed line) stress-strain curves for $\Phi_L = 0^\circ$.

Fig. A.3 Experimental and CPFEM (dashed line) stress-strain curves for $\Phi_L = 45^\circ$.

A.5a, the major part of plastic deformation is accomplished by mixed slip systems as to be expected. Minor activity of longitudinal and transversal systems can be predicted for the last stages of the test, which can be attributed to an increase in the m of these systems due to lattice rotation (see Figure A.5b).

The results dragged from the numerical simulations corresponding to the model with $\Phi_L = 45^\circ$ are presented in a black dashed line in Figure A.3. These results have allowed to match the constitutive model parameters for the slip systems of longitudinal type, with a good fit between experimental and simulations. The fact that FEM modelling is based on a continuous method makes it unfeasible to reproduce the high discontinuities observed experimentally due to dislocation bursts, but the kinematics of deformation are well captured. However, it was possible to fit the envelope of the experimental curves, which is a good approximation for establishing a τ_{CRSS} value for the slip systems of longitudinal type and validate the experimental procedure based on traces observations, described in Chapter 4. Furthermore, the majority of plastic shear is accomplished by longitudinal systems as predicted by the model (see Figure A.5c) and lattice rotation is negligible for this orientation (Figure A.5c), since large strains can be accomplished by the MP as observed experimentally (Section 4.3.1.1).

Fig. A.4 Experimental and CPFEM (dashed line) stress-strain curves for $\Phi_L = 90^\circ$.

Fig. A.5 Normalised accumulated plastic shear and Schmid factor CPFEM prediction for $\Phi_L = 0^\circ$, 45° and 90° .

For the case of $\Phi_L = 90^\circ$, the numerical simulations were utilised to further identify the cause for the multiple activity observed in the micrographs in Figure 4.12. Figure A.5e dragged from a simulation of a model with a lamellar orientation of $\Phi_L = 90^\circ$ predicts how during the evolution of uniaxial compression some longitudinal slip systems acquire a higher m value, which at some point is enough to induce the activation of these systems (Figure A.5f). From this instant on longitudinal slip assumes the majority of the total plastic deformation while transversal systems activity reaches a plateau. Additionally, high levels of hardening are to be expected from the interaction between transversal systems and the interfaces of the lamellar microstructure, which is confirmed through the experimental stress-strain curves.

A.6 Summary

CPFEM has allowed us to confirm the experimental evidence and to define a τ_{CRSS} for each mode of deformation that matches the experimentally measured values through traces analysis described in Chapter 4. Also, the level of activity of each system can be predicted through the study of lattice rotation and the yield asymmetry behaviour for hard loading modes is discussed based on the results.

This work can serve as a first approach for the development of mesoscale plasticity models that capture the micromechanics of fully lamellar TiAl microstructures at larger length scales.

Appendix B

EBSD data processing

Recent developments in EBSD have enabled great improvements in indexing reliability and accuracy, leading to solving the pseudo-symmetric mis-indexing associated to $\gamma(\text{TiAl})$ due to its close tetragonal $c:a$ unit cell parameter ratio of ≈ 1.012 (Sitzman et al.; Zambaldi et al., b). However, although these improvements have allowed an increase in resolution and quality of $\gamma(\text{TiAl})$ EBSD maps, they still lack of consistency when analysing the crystallographic orientation of individual lamellae, being the $\{111\}_\gamma$ planes parallel to the interfaces different for each variant. This is, indeed, caused by the multiple orientation description due to the crystal symmetry, and the applied EBSPs indexing algorithm (Nolze, 2015); deriving in a lack of correspondence between variants within a colony. This fact makes it difficult to conduct a systematic analysis of the deformation systems and their Schmid factor (m) following a morphological based classification criterion, since the same $(hkl)_\gamma[uvw]_\gamma$ system in variant γ_i will belong to a different morphological set in γ_j ($i \neq j$) if both variants do not share their $(111)_\gamma$ pole. This issue is illustrated in Figure 6.5b, where the $(111)_\gamma$ pole figures of the different variants are plotted, according to which only variant γ_3 presents its $(111)_\gamma$ plane parallel to $(0001)_{\alpha_2}$, i.e., parallel to the lamellar interfaces. In Figure 6.5c it is shown the equivalent symmetric solution of interest for each of the remaining variants, with their $(111)_\gamma$ pole perpendicular to the interfaces, obeying the Blackburn relationship for a particularised $(111)_\gamma$ plane. Thus, allowing for a common definition of $(hkl)_\gamma[uvw]_\gamma$ systems among the γ variants coexisting within a colony, sharing the same morphological classification. Furthermore, in the pole figures in Figure 6.5c it is evident that variant γ_1 does not belong to the colony conforming the gage length of the specimen. The average

as-indexed orientation description for each variant and their equivalent symmetrical solution of interest are indicated in Table B.1 in Bunge notation.

Table B.1 Orientation description of γ variants in Bunge notation.

Variant	As indexed			Symmetric solution		
γ_1	75.7°	124.1°	3.3°	75.7°	124.1°	3.3°
γ_2	150.7°	17.4°	35.4°	150.7°	17.4°	215.4°
γ_3	140.3°	127.4°	42.5°	140.3°	127.4°	42.5°
γ_4	94.4°	100.8°	75.8°	94.4°	100.8°	345.8°
γ_5	7.8°	76.3°	78.8°	187.8°	103.7°	101.2°
γ_6	22.0°	122.7°	44.2°	202.0°	57.3°	135.8°

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