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3D characterisation of early twin formation in Ti-4Al by diffraction contrast tomography

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Abstract

Twin formation in α -Ti is a topic of much debate as the role of microstructure features and alloy chemistry on twin formation remains unclear. In the present work, early stage of twin formation, i.e. after 1% plastic compression, has been studied in 3D using diffraction contrast tomography. This technique has enabled to study a volume about 400 grains enabling statistical analysis. The family of twinned grains was compared with similarly orientated grains that had not twinned in respect of their neighbourhood and grain size. While the initial neighbourhood analysis did not identify any significant differences, 3D grain size analysis highlighted that grain size plays an important role during the early stage of twinning. Further, twin clusters/chains were identified, which form slightly imperfect 3D networks. A more detailed neighbourhood analysis of the parent grains associated with the twin clusters/chains demonstrate that strain localization in neighbouring grains well aligned for prismatic $\langle a \rangle$ slip transfer promote the observed twin clustering.

Introduction

Titanium is known to display easy $\{10\text{-}10\}\langle 11\text{-}20 \rangle$ prismatic slip while pyramidal $\langle c+a \rangle$ slip has been observed but only in small fractions [1-3]. This is due to pyramidal $\langle c+a \rangle$ slip having a critical resolved shear stress at room temperature about 3-5 times higher than for prismatic $\langle a \rangle$ slip [4-6]. In the absence of easy slip including a $\langle c \rangle$ component, twinning is commonly observed in Ti and other metals with an hcp crystal structure. This is particularly the case during compression loading [7]. In α titanium, four different twinning modes have been reported [8]. At room temperature, the predominant twinning mode is the $\{10\text{-}12\}\langle -1011 \rangle$ tensile twin [9,10], which corresponds to a rotation of 85° around the $\langle 11\text{-}20 \rangle$ axis. In some cases, this twin mode has been observed to result in almost complete grain reorientation after only modest levels of strain [7,9,11].

The applied stress is expected to play an important role in twin nucleation, as discussed in [3] and [12]. In general, they state that the applied stress is proportional to the number of twins per unit of observed area. To understand the increasing density of twins with increasing levels of applied stress there can be two scenarios: (i) the stress is considered as uniform field applied on an array of potential twin nucleation sites, in which the increasing levels of applied stress allow more sites to be activated; (ii) the critical stress for nucleation is met only at the most potent of the stress concentrations. When increasing the applied stress, more of the available stress concentrators provide stresses that attain the critical level. In most of the cases, the reality is most likely to correspond to a combination of these two scenarios. Localised slip resulting in dislocation pile up at grain boundaries has also been found to play a role in twin nucleation, which has been recently investigated in commercially pure Titanium grade 1 [13]. The slip activity in a grain well aligned for prismatic $\langle a \rangle$ slip leading to

twin nucleation in a neighbouring grain not well aligned for $\langle a \rangle$ type slip was shown to be significant when a tensile stress state was created [13]. Twin to twin shear transfer across grain boundaries has also been observed, particularly in boundaries with misorientations lower than 30° [14]. These correlations were quantified using the slip transfer parameter m' , which was first introduced by Luster and Morris [15].

Considering the importance of neighbourhood in relation to twinning, several synchrotron X-ray techniques might be of particular interest as they enable 3D analysis of polycrystalline materials non-destructively. Examples here are the differential aperture X-ray microscopy (DAXM) [16] and far-field 3D X-ray diffraction (3DXRD) [17]. X-ray DCT [18,19] is a variant of the 3DXRD microscopy technique enabling simultaneous reconstruction of the 3D microstructure (shape and orientation) in suitable polycrystalline materials, along with the absorption map of the specimen. The X-ray DCT methodology provides access to the 3D shape, orientation and elastic strain state of the individual grains from polycrystalline materials fulfilling some conditions in terms of grain size and intra-granular orientation spread.

In the present work the onset of twinning is studied in a binary Ti-4Al alloy that had been compressed to about 1% strain before a small sample was extracted (about 0.04% of the initial sample volume) and characterised using the X-ray DCT methodology to reveal the 3D grain structure, allowing a grain-by-grain study of the shape and location of twins. A statistical analysis was carried out in which the parent grains of twins were grouped together and compared with similarly orientated grains that had not twinned. Comparisons were carried out regarding grain size and slip transfer parameter m' [20] within clusters of parent grains that had twinned and compared with non-twinned grains of similar crystallographic orientation.

Experimental Methodology

For the purpose of this research, 200 g binary Ti-4Al (i.e. 4wt.%) alloy buttons were double melted in a tungsten arc furnace under inert gas atmosphere. This was followed by beta forging at 1100°C at the TIMET - Savoie research facility in Witton, UK.

The measured chemical composition of the alloy is given in Table 1. Subsequently, the buttons were cross-rolled in bar shape ($14 \times 14 \times 260$ mm) on a “2 high Robertson mill” (WHA Robertson & Co Ltd) at 870°C followed by a recrystallization (RX) heat treatment at 993°C (30°C below the beta transus temperature) in a tube furnace under Argon shielding for 5 hours followed by air-cooling. The average grain size of the studied samples is $73\text{ }\mu\text{m}$, which was measured by using the linear intercept method. The $\{0002\}$ pole figure and the initial microstructure of the Ti-4Al raw material used in this analysis is shown in Figure 1.

Table 1: Chemical composition of Ti-4Al:

Al	C	N	O	Ti
3.9 wt.%	50 ppm	23 ppm	778 ppm	bal.

Three samples with a diameter of 5 mm and height of 12.35 mm each were cut by electro-discharge machining with the cylinder axis parallel to the original rolling direction (RD). The texture of the material is such that the $\langle c \rangle$ axes of the grains tend to be oriented perpendicular to the cylinder axis, which promotes tensile twinning during compression loading as the $\langle c \rangle$ axis is strained in tension.

Figure 1: Initial microstructure of the material represented in terms of (a) $\{0002\}$ pole figure and (b) grain orientation map recorded by EBSD.

After deformation of a standard compression sample, a sub-scale sample of about 600 μm diameter and about 1.5 mm height was extracted and studied by X-ray DCT at beam line ID11 of the European Synchrotron Radiation Source (ESRF), Grenoble, France, using a monochromatic beam produced by a bent Si 111 Laue-Laue double-crystal monochromator (40 keV, wavelength $\lambda = 0.309 \text{ \AA}$, relative bandwidth $\Delta\lambda / \lambda \sim 10^{-3}$). A high resolution detector system consisting of a FReLoN charge-coupled device (CCD) camera equipped with a scintillator screen and visible light microscope optic was used for this experiment, positioned normal to the incident beam, about 5 mm downstream from the sample. The detector has an array of 2048×2048 pixels² with an effective pixel size of 1.4 μm and an active area of $2.87 \times 2.87 \text{ mm}^2$. An absorber was inserted between the sample and the detector to attenuate the transmitted beam without affecting the diffracted beams. This allows the integration time per image to be increased in order to improve the intensity particularly of weak diffraction spots (i.e. the ones related to twin lamellae).

The standard X-ray DCT analysis methodology has previously been described in detail [18,19]. A series of images (typically 3600-7200) are recorded while the sample is rotated through 360° . The background is subtracted from the images, leaving only the diffraction spot images. These are segmented, and metadata are recorded that describe the spots. The geometry of diffraction events is extracted using a Friedel pair geometry, based on identifying pairs of spots arising from the scattering vector and same grain, offset by 180° rotation of the sample. The Friedel pair construction reveals the diffraction angles and scattering vector associated with the diffraction event, and a path through the sample on which the grain must lie. Grain positions and orientations are identified by searching for Friedel pairs that are spatially and crystallographically consistent. The grain shapes are then reconstructed using a simultaneous iterative reconstruction technique (SIRT) algorithm, using the diffraction spot images as grain projections. Subsequently, the grains shapes

are assembled to produce a 3D grain map. Any overlapping or unassigned spaces in the 3D map are filled using a morphological dilation. The complete procedure of data processing can be found in [18,19].

An iterative tomographic reconstruction process was performed using an implementation of the SIRT algorithm available in the All Scale Tomographic Reconstruction Antwerp (ASTRA) tomographic toolbox. The process assumes that all grains can be indexed in the volume and remaining gaps in this initial grain map are removed by dilating the existing grains until a space filling grain map is obtained.

Figure 2: 3D grain map of the reconstructed sample volume coloured according to the IPF colour code for hcp materials (c). Full 3D grain maps of (a) all the grains and (b) only twinned grains are displayed.

An absolute measure of the illuminated sample volume ($V_{\text{sample}} = 0.0757 \text{ mm}^3$) was obtained from the tomographic reconstruction of the transmission images recorded during the scan. In order to identify the expected $\{10\text{-}12\}\langle\text{-}1011\rangle$ tensile twins an automated search was carried out to identify grains overlapping in space and fulfilling the orientation requirements of any of the twinning modes for hexagonal titanium before the $\{10\text{-}12\}\langle\text{-}1011\rangle$ tensile twinning mode was selected (the angle between the $\langle c \rangle$ axis of the two grains must be 85° and the rotation axis parallel to one of the six $\langle 11\text{-}20 \rangle$ directions).

One particular point of interest in the analysis of the volume was the Luster-Morris parameter, which can be used to describe relative alignment of neighbouring grains in respect of certain planes and directions. It is defined by equation (1):

$$m' = \cos \psi \cos \kappa \quad (1)$$

where ψ is the angle between two plane normals and κ is the angle between the associated slip/shear directions. Hence, $m' = 1$ indicates perfect alignment of two neighbouring grains for easy slip transfer across the grain boundary. In the present work the Luster-Morris parameter was considered for neighbouring grains aligned for prismatic to prismatic slip transfer and $\{10\text{-}12\}\langle\text{-}1011\rangle$ twinning shear to $\{10\text{-}12\}\langle\text{-}1011\rangle$ twinning shear transfer between neighbouring grains.

Results

The reconstructed volume of the X-ray DCT scan, together with the volume of the twinned grains only, is presented in Figures 2a and 2b. In both figures grains are coloured according to the inverse pole figure scheme with the sample loaded along (0001), Figure 2c.

The reconstructed grain volumes were dilated using a mask determined from absorption tomography in order to fill the empty spaces between the grains (a single grain dilation of maximum 7 μm has been used). The reconstructed sample volume, resulting from concatenation of 3 DCT scans, resulted in a height of 0.77 mm and a volume of about 0.0757 mm^3 . The total number of grains in the studied volume is 402, out of which 58 have twinned (some of them containing multiple twins) and 67 of the 70 indexed twins have been identified as $\{10\text{-}12\}\langle\text{-}1011\rangle$ tensile twins (~17% with respect to the total number of indexed grains). The crystallographic orientations of the 402 grains are presented in form of an inverse pole figure map in Figure 3a, distinguishing between twinned grains, twins and grains that did not twin. It can be stated that the twinned grains (squares) are distributed uniformly in the region between (10-10) and (2-1-10) poles. Figure 3a also highlights that the studied volume contains plenty of grains without twins that are similarly orientated to the grains that have twinned. In order to compare twinned and non-twinned grains in a meaningful way, each twinned grain was paired with the most similarly orientated grain that had not twinned, as shown in figure 3b. This was achieved by calculating a distance in the orientation space between individual twinned grains and non-twinned grains. The orientation space is defined by two angles, the azimuthal angle ϕ and the polar angle ψ , which both describe the inverse pole figure. In order to consider a non-twinned grain of similar crystallographic orientation to a twinned grain the distance d between a twinned grain with coordinates (ϕ_i, ψ_i) and a grain that had not twinned with coordinates (ϕ, ψ) had to satisfy:

$$d = \phi_i - \phi + \psi_i - \psi < 1.5^\circ \quad (2)$$

This tight criterion was chosen to ensure that single solutions could be found for each twinned grain. Following this procedure, 43 similarly oriented grains were found corresponding to twinned grains over a total of 58 twinned grains. Hence, 15% of the twinned grains could not be paired with a non-twinned grain.

The analysis of the twinned grains revealed that 96% of the characterised deformation twins in the sample were $\{10\text{-}12\}\langle\text{-}1011\rangle$ tensile twins, i.e. 67 $\{10\text{-}12\}\langle\text{-}1011\rangle$ tensile twins, one $\{11\text{-}21\}\langle\text{-}1\text{-}126\rangle$ tensile twin and two $\{11\text{-}22\}\langle\text{-}11\text{-}23\rangle$ compression twins.

In a first step of comparing the twinned grain family with the one that had not twinned but was of comparable crystallographic orientation, Figure 4 shows the grain volumes after dilation. It should be noted that the volume of a twinned grain combines the parent grain and the twin volume. Figure 4 clearly demonstrates that the grain volume distribution of the twinned grain family is shifted to a larger volume than the grains without twins. Hence, the grain volume analysis suggests that during the very early stage of plasticity grain size is an important factor for twin nucleation and growth.

During the next stage, the grain neighbourhood of the two grain families was investigated in greater detail. First of all it is important to note that during compression loading and considering the starting texture, the grains that are most likely to twin are also well aligned for deformation by prismatic $\langle\text{a}\rangle$ slip. Hence, a neighbourhood that displays a high Schmid factor of similar

orientation is more likely to allow the twinning grain to build up a high dislocation density and large intergranular strains, i.e. large tensile strains along the $\langle\text{c}\rangle$ axis perpendicular to the compressive loading axis. Both these aspects could be considered to promote $\{10\text{-}12\}\langle\text{-}1011\rangle$ twin nucleation. Figure 5a plots the prismatic $\langle\text{a}\rangle$ slip Schmid factor distribution computed for the grain neighbourhood of both grain families. It can be seen that both grain families display a high fraction of grain neighbours well aligned for prismatic $\langle\text{a}\rangle$ slip but there is no noticeable difference in Schmid factor distribution between the two.

Figure 3: Inverse Pole Figure (IPF) representation of the grain orientations with respect to the sample loading direction (001). In (a), all grain and twin are shown while in (b) only the two grain families, i.e. the twinned grains and their non-twinned counterparts are shown.

Figure 4: Cumulative plot of the grain volume for the twinned grains (squares) and the similarly oriented grains to the twinned grains (circles), calculated from the reconstructed volume after dilation. The grain size is the diameter of the equivalent sphere corresponding to the grain volume and it is shown in the upper horizontal axis. A log scale in base 3 is used for both the horizontal axes.

Figure 5: (a) Schmid factor m for prismatic slip and (b) slip transfer parameter m' for prismatic slip to prismatic slip has been calculated for all the neighbours of the twinned grains (squares) and the similarly oriented grains to the twinned grains (circles).

In addition, figure 5b exhibits the distribution of the transfer parameter m' for prismatic $\langle a \rangle$ slip computed from the neighbourhood of the two different grain families. It is noticeable that both grain families display a wide distribution of m' but the analysis again does not yield a clear differentiation between the twinned grains and the grains that have not twinned.

As a next step, a detailed analysis of the position of the twinned grains was carried out within the studied volume with the purpose of identifying potential chains or clusters of twinned grains and perform a neighbourhood analysis on these. The individual twin chains/clusters are rendered in figure 6a using a different colour for each chain. For clarity, figures 6b – 6h also highlight each individual chain. As this search indicated predominantly clusters rather than chains, the exact identification presented some difficulties. In this case a given twinned grain can have more than two neighbouring twinned grains, which makes the rendering of the neighbourhood more difficult. Each cluster was defined by starting from a grain with only one neighbouring twinned grain and the rendering did proceed by choosing the grain with the lowest number of neighbours among the neighbouring twinned grains, in order to simplify the choice as much as possible. As such search needs to be carried out in 3D space the calculation and the identification of the clusters was very computing intensive.

Among 58 twinned grains, 7 different clusters/chains were identified with the shortest cluster consisting of 3 twinned grains and the largest cluster containing 14 grains. In general, the twins linked up well within a cluster as demonstrated in figures 6b – 6h. In addition, only 3 isolated twinned grains were identified. It is also worth noting that a careful analysis of all the clusters/chains revealed that they seem to have formed a slightly imperfect network, i.e. only small gaps are present between the clusters/chains.

Figure 6: (a) 3D rendering of the identified chains of twinned grains. (b)-(h) Each chain is visualised with a different colour and displayed in a separate figure. The darkest colour has been assigned to the first grain of the chain. (i) Sample reference system defined by x-axis (red), y-axis (green) and z-axis (blue).

Figures 7a and 7b display the distributions of both transfer coefficients computed from within the chains/clusters and compare them with the previously computed transfer coefficient between the twin-free grain family and their neighbourhood. Such more local comparison reveals that within clusters/chains the grains are particularly well aligned for transferring prismatic $\langle a \rangle$ slip showing significantly higher m' values compared to the twin-free grain family, figure 7a. In contrast, the m' coefficient of $\{10-12\} \langle -1011 \rangle$ tensile twinning within twin clusters/chains remains very similar to the twin-free grain family, figure 7b.

Discussion

The results presented here clearly demonstrate that X-ray based diffraction contrast tomography is an excellent tool to undertake detailed analysis of twinned grains and their neighbourhood. It should be kept in mind that twins are detected by capturing the diffraction spot signals of the twin. Clearly, in the case of very thin twins, there is a high possibility that a twin is missed in the analysis. The thinnest twin detected in the present case was about

14 μm wide and occupied a volume of $1.2 \cdot 10^3 \mu\text{m}^3$. Hence, one might assume that the present analysis captures the twins that formed first and have grown to a size that has made them detectable. An important aspect of the present work is that a sufficient number of twins were captured to undertake a statistical analysis of the neighbourhood and compare with similarly orientated grains that had not twinned. This analysis reveals a number of important drivers for early twin formation and that there is a tendency for the formation of twin clusters and chains, which seem to develop into a twin network, Figure 6.

Figure 7: Distributions of transfer parameter m' for (a) prismatic slip to prismatic slip and for (b) $\{10-12\}\langle-1011\rangle$ tensile twin to $\{10-12\}\langle-1011\rangle$ tensile twin for all the twinned grains of the chains (squares) and all the neighbours of the similarly oriented grains to the twinned grains (circles). The twinned grains are excluded from the neighbours of the similarly oriented grains.

Regarding drivers for early twin formation, the 3D nature of the analysis has revealed the importance of grain size for early twin formation, figure 3. While positive grain size dependency of twinning has been reported previously for Mg [21], Ti [22] and Zr [7], the present 3D analysis provides a very clear picture of the size dependency because it does not suffer from intrinsic broadening of the distribution as a result of a 2D analysis. It has been suggested previously that the root cause for less twin formation in the case of small grains is related to the twin boundary energy cost being relatively high compared to the plastic work induced by the twin [23]. It has also been suggested that the size effect is simply related to a decrease in the number of twins with decreasing grain size assuming a constant twin per unit of grain boundary ratio [24]. This consideration assumes the importance of grain boundaries on twin nucleation. The true twin thickness has been found to be independent of grain size in

magnesium and zirconium by Tome' and colleagues [7,25] but strongly dependent on the grain size in titanium [26]. Nevertheless, they found that the number of twins, in grains that did twin, increased with increasing grain size. Armstrong and Worthington [27] suggest that twinning is associated with micro-plasticity and high stress concentrations within local regions. As X-ray DCT might not capture the very early stage of twin formation, the observed grain size dependency could indicate easier growth of twins in comparatively large grains.

An important factor regarding twin nucleation is also the stress acting on the grains during mechanical loading. It has been demonstrated previously that compression loading of Ti and Zr, with similar starting textures as in the present case, first result in deformation by slip before twinning is observed [28,29]. In this configuration, it is the 'soft' grains, i.e. grains well aligned for prismatic $\langle a \rangle$ slip, which are prone to form $\{10-12\}\langle-1011\rangle$ tensile twins in order to relieve the tensile stress along the c -axis generated in the transverse direction. It is also the configuration that tends to result in the highest twin activity when the material is deformed. Hence, one might argue that prismatic $\langle a \rangle$ slip activity is a critical parameter in order to induce sufficiently large intergranular strain along the $\langle c \rangle$ axis to nucleate $\{10-12\}\langle-1011\rangle$ tensile twins.

It should be noted that in a polycrystalline aggregate, the level of plasticity experienced by a grain does not necessarily depend on the global but the local stress state. For instance, a grain well orientated for prismatic $\langle a \rangle$ slip in respect of the applied loading direction, but surrounded by grains well aligned for pyramidal $\langle c+a \rangle$ slip (hard orientation), will be shielded and is unlikely to experience high levels of plasticity. In contrast, if neighbouring grains are well aligned for prismatic slip, and prismatic $\langle a \rangle$ 'slip transfer' across grain boundaries is possible, high levels of plasticity are likely in those grains. This will produce high intergranular strains, which could be a driver for twin nucleation. For this reason, the prismatic $\langle a \rangle$ slip transfer analysis is of importance here. Recent crystal plasticity prediction have indeed suggested for Mg that strain localisation, in this case due to basal $\langle a \rangle$ slip, does result in intergranular stress concentration in those grains, which promote twin formation and clustering [30]. Figure 7a clearly displays a shift to higher values of the m' -value distribution, computed from within twin clusters/chains compared to the m' -values of the non-twinned grain family in respect of its neighbourhood, Figure 7a. Hence, the evolution of twin chains or clusters seems to be guided by following a chain/cluster of grains that can be linked by high prismatic $\langle a \rangle$ slip m' -values, i.e. a cluster of parent grains being well aligned for deforming first by prismatic $\langle a \rangle$ slip. In contrast, a similarly sophisticated analyses of the transfer coefficient for $\{10-12\}\langle-1011\rangle$ tensile twinning did not display a difference of values computed from within twin chains/clusters compared to calculating the transfer coefficient distribution of the related twin-free grain family and their neighbourhood, Figure 7b. This is interesting as one might argue that a shift towards higher m' -values within twin clusters/chains would have emphasised the importance of twins nucleating twins in neighbouring grains by creating stress concentrations, as observed in [14] {Barnett 2012}. As this concept should be most effective when the shear strain of the twinned grain is of similar direction of the twin shear strain induced in the neighbouring grain, i.e. high m' -value, the observations in figure 7b suggest that this mechanism only plays a minor role in the formation of twins in a polycrystalline aggregate with the given crystallographic texture.

Finally, it is worth emphasising that the present observations are relevant for compression loading and that the situation during tensile loading will be a very different one as in such case the $\{10-12\}\langle -1011 \rangle$ tensile twins form in grains that are not well aligned for any easy $\langle a \rangle$ slip [13].

Conclusions

A detailed analysis of a Ti-4Al sample compressed along the former rolling direction by only 0.8% plastic strain has been carried out first by using in-situ loading and neutron diffraction on SALSA, ILL, followed by X-ray Diffraction Contrast Tomography (DCT) on ID11, ESRF. The main purpose of this work was to study the onset of deformation twinning and consider the true grain neighbourhood in this 3D analysis in order to elucidate possible twin nucleation criteria. An important aspect of the analysis was to compare the family of twinned grains with a family of similarly orientated grains that had not twinned yet and identify key differences. The main findings can be summarised as follows:

- The neutron diffraction analysis enabled the early detection of $\{10-12\}\langle -1011 \rangle$ tensile twinning, which was seen after less than 1% plastic strain. A subsequent X-ray DCT analysis confirmed the dominance of $\{10-12\}\langle -1011 \rangle$ twins.
- The X-ray DCT analysis enabled the identification of almost 60 twinned grains with a few cases of multiple twins per grain within a sample volume of about 400 grains. It is important to note that it is possible that only twins that formed very early and have grown to a appreciable size were captured.
- A grain volume/size distribution analysis comparing twinned grains including the twin volume and similarly orientated grains that had not twinned revealed a clear shift towards a higher mean value in the case of the twinned grain family highlighting the importance of grain size on twin formation.
- An initial statistical analysis of the neighbourhood comparing the family of twinned grains with the crystallographically related non-twinned grain family did not reveal any significant differences.
- The 3D analysis revealed chains and clusters of twins that have also developed a slightly imperfect twin network. This suggests a strong neighbourhood effect.
- A Luster-Morris parameter analysis in respect of prismatic $\langle a \rangle$ slip and twin shear within chains/clusters and in comparison to the neighbourhood of the non-twinned grain family revealed that the twin clustering is most likely related to easy prismatic $\langle a \rangle$ slip transfer between the parent grains that form the twin chains/clusters. In principle, this finding is in good agreement with [30], which predicts the formation of twin clusters and the role of $\langle a \rangle$ slip strain location in Mg. In contrast, easy shear transfer from $\{10-12\}\langle -1011 \rangle$ twins was not identified as important factor within the chain/clusters suggesting that twin nucleation by stress concentration from a neighbouring grain that has twinned is not a main driving force for twin formation.

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