Nanostructure formation mechanism of $\alpha$-titanium using SMAT

K.Y. Zhu $^a$, A. Vassel $^b$, F. Brisset $^c$, K. Lu $^d$, J. Lu $^{a,*}$

$^a$ LASMIS, FRE CNRS 2719, University of Technology of Troyes, 12 Rue Marie Curie, BOP 2060-10010 Troyes Cedex, France
$^b$ Materials Systems and Composites Department, ONERA, BP 72, 92322 Châtillon Cedex, France
$^c$ LEM-CNRS, ONERA, BP 72, 92322 Châtillon Cedex, France
$^d$ Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016, People’s Republic of China

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Abstract

A nanostructured surface layer up to 50 $\mu$m thick was produced on commercially pure titanium using surface mechanical attrition treatment (SMAT). The microstructural features of the surface layer produced by SMAT were systematically characterized by cross-sectional optical microscopy observations, transmission electron microscopy (TEM) and high-resolution transmission electron microscopy (HRTEM) investigations. The grain refinement process, accompanied by an increase in strain in the surface layer, involves: (1) the onset of twins and the intersection of twin systems, (2) the formation of dislocation walls, (3) the nucleation of microbands associated with the splitting of dislocation walls, (4) the subdivision of microbands into low angle disoriented blocks and then highly disoriented polygonal submicronic grains, and (5) further breakdown of submicronic polygonal grains into randomly oriented nanograins. The final grain refinement step to form nanograins has been discussed on the basis of a recrystallization process.

E-mail addresses: lu@utt.fr, jian.lu@utt.fr (J. Lu).

1. Introduction

Nanocrystalline and submicrocrystalline materials have been the subject of extensive investigations during the past decade owing to their considerable scientific and practical interest [1]. Studies of grain refinement mechanisms in metals and alloys represent one of the most advanced directions in physical metallurgy. The resulting fine grain structure provides a significant increase in both the low and high temperature strength [2] and the tribological properties [3].

Various processing routes have been developed to produce bulk nanocrystalline materials, e.g. the consolidation of ultrafine powders produced using different techniques [4], the crystallization of amorphous precursors [5], electrodeposition [6] and severe plastic deformation of bulk materials [7]. However, two major obstacles still impede the development of bulk nanocrystalline materials: (i) it is difficult to manufacture porosity-free and contamination-free samples, and (ii) most of the current techniques for synthesizing bulk nanocrystalline materials are not adapted to production on an industrial scale due to limitations in terms of sample size and cost.

Optimization of the surface structure of materials is of great concern at the present time since most failures occur on the surface (fatigue, fretting corrosion, corrosion, wear, etc.). As a result, improving the surface properties would greatly enhance the overall behavior of materials. The importance of the surface structure and the attractive properties afforded by nanostructured materials have led to the development of a new concept called surface nanocrystallization (SNC) [8]. It has been demonstrated that among the different techniques used to produce nanostructured layers, surface mechanical attrition treatment (SMAT) is an effective way of creating localized plastic
deformation, resulting in grain refinement down to the nanometer scale without changing the chemical composition of the material [9]. The SMAT process has been successfully applied to various material systems including aluminum [10], iron [11,12] and stainless steel [13,14]. Another advantage of surface nanocrystallization is that it greatly enhances the diffusion kinetics of atoms. It has been found that the nitriding temperature of iron produced using SMAT iron can be reduced to 300 °C, which is at least 200 °C below the conventional nitriding temperature [15].

The nanocrystallization of titanium and its alloys was first studied in the early 1990s with the aim of producing bulk materials. Two main processing methods were used, i.e. mechanical alloying [16] and magnetron sputtering [17]. Several titanium systems were also considered such as Ti–Mg, Ti–Ni, Ti–Cu, Ti₃Al–Nb and TiAl [16–20]. Despite the various efforts made, development of the above materials has been hindered by interstitial contamination (O, N, H) during fabrication and the fact that only very small specimens can be produced.

Recent investigations have demonstrated that commercially pure titanium with an ultrafine grain structure in the nanometer range can be processed using severe plastic deformation methods such as equal channel angular pressing (ECAP) and high pressure torsion (HPT) [21–25]. One advantage of these methods is that they produce samples that have no porosity, which makes it possible to produce meaningful measurements of their physical and mechanical properties. It has been shown that these new processes nearly triple the strength of pure titanium, which could have a worldwide impact on future applications of this material [23].

It is widely recognized that titanium displays poor wear resistance and that its fatigue performance depends to a large extent on its surface properties. The newly developed SMAT process is therefore of considerable technological importance since it provides the possibility of dramatically improving the surface properties of titanium. As a result, extended applications which demand high levels of reliable performance can be anticipated in surgery and medicine as well as in the aerospace, automotive, chemical plant, power generation and other major industries.

The present work aims at studying the grain refinement mechanism which occurs in commercially pure titanium during the SMAT process. The application of SMAT to titanium is of interest from an academic point of view because it has an hcp structure, whereas all the metals studied up to now have been cubic (Al, Fe, Cu, Ni). It is also obvious that the deformation modes (twinning, slip) have a considerable influence on the grain refinement mechanism. In Al (fcc) and Fe (bcc), for instance, dislocation glide is the main deformation mode and it has been found to dominate the grain subdivision process down to the nanometer scale when SMAT is carried out [10,11]. In other metals such as stainless steel, dislocation glide is the prevalent deformation mechanism at low strain and is converted into twinning as the strain increases [13]. In contrast to this, however, results obtained for cold working deformation of titanium show that twinning occurs first, and that dislocation slip takes over at high strain levels [26].

2. Experimental methods

The material used in this investigation is an annealed, commercially pure titanium bar 50 mm in diameter, with the following chemical composition (wt%): 0.010 C, 0.035 Fe, 0.105 O, 0.005 N and 0.001 H. Its microstructure consists of equiaxed 20–50 μm α grains. Prior to SMAT, 10 × 20 × 20 mm³ parallelepipedic specimens machined from the bar were surface-polished with silicon carbide paper to grade 1200.

The SMAT set-up and procedure have been described in detail elsewhere [27]. Fig. 1 gives a schematic illustration of the SMAT process. The technique of the present work is based on the vibration of spherical shots using high-power ultrasound. Because of the high frequency of the system (20 kHz), the entire surface of the component to be treated is peened with a very high number of impacts over a short period of time. The approximate strain rate is 10²³ s⁻¹ near the top treated surface. The SMAT was performed in air at room temperature using a prototype SNC-2 machine. The different SMAT conditions selected are shown in Table 1.

A Reichert MeF3 optical microscope was used to examine the microstructural development along sections perpendicular to the treated surface of the specimens. The cross-sections were mechanically polished using silicon carbide paper to grade 4000, then on a polishing cloth with a liquid suspension of 0.04 μm alumina, and finally etched at room temperature in a solution of 2 ml HF, 3 ml H₂O₂ and 96 ml distilled water.

![Fig. 1. Schematic illustration of the SMAT technique.](image-url)
Transmission electron microscopy (TEM) investigations were carried out on JEOL-4000FX and JEOL-200CX microscopes operating at 400 and 200 kV, respectively. High resolution TEM was performed on the JEOL-4000FX microscope. The plane-view TEM foils of layers at different depths were obtained first by polishing the corresponding surface layer, then mechanically polishing the sample on the untreated side until it was about 30 μm thick; it was then electro-polished using a twin-jet technique in a solution of 590 ml CH₃OH, 350 ml C₆H₁₄O₂ and 60 ml HClO₄ at a voltage of 23–25 V and a temperature of -40 °C.

3. Results

3.1. Optical microscopy observations

The cross-sectional optical microstructures of the SMAT-treated specimens under different conditions are shown in Fig. 2. It can be seen that the deformation layers produced under different treatment conditions have different features. When treatment is carried out at low amplitude (50%) for a short time (10 min), a deformation layer can be observed consisting of many mechanical twins which become more and more dense as they approach the treated surface. Intersections of twins are also visible in the top layer of the sample (Fig. 2(a)). When the amplitude (100%) and time (16 min) are increased, the deformed layer becomes thicker and a layer of very fine lamellae forms close to the top surface (Fig. 2(b)). If the time is extended to 30 min, a submicronic structure appears close to the top surface which is outside the resolution of optical microscopy (Fig. 2(c)). After one hour, the thickness of the submicronic and fine lamellar structure layer increases (Fig. 2(d)). It can be noted that the gradient structure resulting from a gradual decrease in the applied strain and strain rate as the depth of the treated surface layer of specimens N3 and N4 increases, from very high (top surface) to zero (substrate), represents the complete range of structural changes which occur during treatment. These specimens were therefore selected to investigate the grain refinement mechanism by examining the microstructural features at different depths in the deformed surface layer. Based on the optical micrographs of specimens N3 and N4, depths of about 20, 60, 150 and 300 μm from the SMAT-treated surface were selected for investigation by TEM.

<table>
<thead>
<tr>
<th>Specimen code</th>
<th>Frequency (kHz)</th>
<th>Diameter of stainless steel shot (mm)</th>
<th>Vibration amplitude (%)</th>
<th>Time (min)</th>
</tr>
</thead>
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<tr>
<td>N1</td>
<td>50</td>
<td>10</td>
<td>20</td>
<td>100</td>
</tr>
<tr>
<td>N2</td>
<td>20</td>
<td>3</td>
<td>100</td>
<td>16</td>
</tr>
<tr>
<td>N3</td>
<td>100</td>
<td>30</td>
<td>100</td>
<td>30</td>
</tr>
<tr>
<td>N4</td>
<td>60</td>
<td>60</td>
<td>100</td>
<td>60</td>
</tr>
</tbody>
</table>

![Fig. 2. Cross-sectional optical micrographs close to the SMAT-treated surfaces under different conditions: (a) N1; (b) N2; (c) N3; (d) N4.](image-url)
3.2. TEM investigations

3.2.1. 300 μm below the treated surface

The microstructure of specimen N3 consists of parallel or interlaced bands with widths varying from several tenths of a nanometer to a few microns (Fig. 3(a)). Selected area electron diffraction (SAED) patterns taken from areas containing two adjacent bands reveal that they are twins. Fig. 3(b), for instance, illustrates a SAED pattern of this type showing a twin relationship; mirror spots can be observed with respect to the \( \{1012\} \) plane of the \( \alpha \) phase, indicating a \( \{1012\} \) twin system. The twins generally run across the entire length of the grain and stop at the boundary, but occasionally, one of the ends terminates inside the grain. When this happens, the ends of the twins are lenticular in shape and taper to a sharp edge at the tip (Fig. 3(a)), as the result of a shear accommodation process [28].

When the treatment time is increased (N4), thereby causing greater strain, a larger number of parallel and intersected bands can be observed (Figs. 4(a) and (b)) at the same depth from the treated surface. Their boundaries are not as straight as those of the twins in N3 and a lot of dislocation pile-ups are present on or near the boundaries (Fig. 4(c)). Twin relationships between these bands cannot be identified using SAED, but a disorientation of about several degrees can be observed (Fig. 4(d)). These lamellae may represent the evolution of twins observed in the specimen at a deeper layer.

3.2.2. 150 μm below the treated surface

TEM examination of the layer about 150 μm below the surface of specimen N4 shows that the microstructure differs considerably from that observed at a depth of 300 μm. Very fine parallel lamellae or microbands (100–300 nm in thickness), most of which are divided into a series of blocks (Figs. 5(a) and (b)), exist almost everywhere. In some places, the fine lamellar structure has virtually disappeared and has been converted into 200–400 nm submicronic polygonal grains (Fig. 5(c)). SAED patterns in areas with a diameter of 7 μm show arcing spots indicative of disorientation between adjacent polygonal grains (Fig. 5(d)). It is reasonable to believe that the increase in strain is the cause of the subdivision of lamellae into blocks and the formation of polygonal grains.

3.2.3. 60–70 μm below the treated surface

Observation of specimen N4 shows scattered areas of residual lamellae or polygonal grains similar to those described above. The residual lamellae contain a high

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Fig. 3. (a) Bright field image of parallel and intersected twins in the area adjacent to the substrate; (b) corresponding SAED pattern and indexation.

Fig. 4. (a) Bright field image of parallel and intersected twins in the area adjacent to the substrate; (b) corresponding SAED pattern and indexation.

Fig. 5. (a) Bright field image of parallel and intersected twins in the area adjacent to the substrate; (b) corresponding SAED pattern and indexation.
Fig. 4. (a,b) Bright field images of interlaced lamellae; (c) bright field image of dislocation pile-ups on or near lamellae boundaries; (d) SAED pattern showing a disorientation of $6^\circ$ between two adjacent lamellae.

Fig. 5. (a) Dark-field image of fine lamellae and blocks; (b) bright field image of blocks; (c) bright field of submicronic polygonal grains; (d) corresponding SAED pattern of (c).
Fig. 6. (a) Dark-field image showing equiaxed nanograins; (b) corresponding SAED pattern of (a); (c) histogram of grain size distribution.

Fig. 7. (a) Dark-field image showing uniformly distributed nanograins; (b) corresponding SAED pattern; (c) histogram of grain size distribution.
density of dislocations. The microstructure is composed mainly of 100–300 nm equiaxed nanograins (Fig. 6(a)). SAED patterns in areas with a diameter of 7 \(\mu\)m exhibit well-defined rings which show that the nanograins are highly disoriented with respect to each other (Fig. 6(b)). The histogram of grain size distribution obtained from dark field images is shown in Fig. 6(c); the grain size corresponds to the Feret diameter of the grains. The histogram is calculated from a single bright field image associated with several dark field images (at least four) in order to determine the size of each grain with greater precision. This method was used because of the variation in the contrast of the grains due to lattice deformation. It is suggested that the nanograins result from the further fragmentation of larger grains and residual lamellae.

### 3.2.4. Top treated surface

An examination of the top surface layer of specimen N4 (about 15–30 \(\mu\)m below the treated surface) shows that the microstructure is characterized by uniformly distributed nanometer-scale grains (Fig. 7(a)). The corresponding SAED patterns of areas with a diameter of 7 \(\mu\)m exhibit more pronounced rings than those at a depth of 60–70 \(\mu\)m (Fig. 7(b)), which illustrates the formation of finer grains with a more random orientation on the top treated surface. The histogram of the grain size distribution of this layer (Fig. 7(c)), calculated using the same method as that described above, shows a grain size of 50–250 nm with a mean value of 150 nm. The question arises as to the nature of the phase formed under severe plastic deformation during SMAT. The diffraction rings in Fig. 7(b) have therefore been indexed and all of them are shown to correspond to the main crystallographic planes of the \(\alpha\) phase (Table 2).

### 3.3. Summary of microstructural observations

Table 3 compares the results of TEM examinations performed at selected depths below the treated surface on N3 and N4 specimens. From a general point of view, it can be seen that the differences in the microstructural features of the two specimens reflect the higher strain in specimen N4 consecutive to a longer treatment time. It was also observed that a very similar nanostructure layer forms on the top treated surface of both specimens.

It is worth mentioning that combining TEM and optical microscopy observations can provide a rough estimation of the thickness of the nanostructure layer which, in fact, corresponds to the layer outside the resolution of optical microscopy observed in Figs. 2(c) and (d), i.e. a thickness of about 40 and 50 \(\mu\)m for specimens N3 and N4, respectively.

### 4. Discussion

Based on the microstructural features observed at various depths and different levels of strain in the deformed surface layer, it can be concluded that, as the strain and strain rate increase, the following changes occur in the microstructure of titanium during SMAT, the details of which are discussed below:

1. The onset of twins and the intersection of twin systems.
2. The formation of low angle disoriented lamellae displaying a high density of dislocations.
3. The subdivision of microbands into blocks and the resulting formation of polygonal submicronic grains.
4. The further breakdown of submicronic polygonal grains into nanograins.

### 4.1. Onset of twins and the formation of lamellae

Five independent slip systems are necessary for polycrystalline materials to undergo homogeneous

<table>
<thead>
<tr>
<th>Depth below surface ((\mu)m)</th>
<th>N3</th>
<th>N4</th>
</tr>
</thead>
<tbody>
<tr>
<td>15–30</td>
<td>Equiaxed nanostructure with a grain size of 50–250 nm. SAED patterns exhibit very well-defined rings</td>
<td>Same as N3</td>
</tr>
<tr>
<td>60</td>
<td>Hybrid microstructure consisting mainly of equiaxed nano-grains 100–300 nm in size and scattered areas of residual lamellae or polygonal grains. SAED patterns exhibit rings</td>
<td>Higher density of nanograins than for N3. SAED patterns show more defined rings than for N3</td>
</tr>
<tr>
<td>150</td>
<td>Lamellae divided into blocks and high angle disoriented submicronic polygonal grains. SAED patterns exhibit dotted spots</td>
<td>Larger areas of submicronic polygonal grains than in N3. SAED patterns exhibit arcing spots</td>
</tr>
<tr>
<td>300</td>
<td>Parallel and intersected twins</td>
<td>Interlaced and low angle disoriented lamellae</td>
</tr>
</tbody>
</table>

**Table 2**

<table>
<thead>
<tr>
<th>Ring</th>
<th>Diameter of ring (mm)</th>
<th>(d) (Å)</th>
<th>Plane</th>
</tr>
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<tbody>
<tr>
<td>1</td>
<td>21.4</td>
<td>2.57</td>
<td>{10,01}</td>
</tr>
<tr>
<td>2</td>
<td>23.2</td>
<td>2.37</td>
<td>{00,02}</td>
</tr>
<tr>
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<td>24.4</td>
<td>2.25</td>
<td>{10,11}</td>
</tr>
<tr>
<td>4</td>
<td>31.8</td>
<td>1.73</td>
<td>{10,12}</td>
</tr>
<tr>
<td>5</td>
<td>37.2</td>
<td>1.48</td>
<td>{11,20}</td>
</tr>
<tr>
<td>6</td>
<td>41.2</td>
<td>1.33</td>
<td>{10,13}</td>
</tr>
<tr>
<td>7</td>
<td>43.2</td>
<td>1.27</td>
<td>{02,20}</td>
</tr>
<tr>
<td>8</td>
<td>44.6</td>
<td>1.23</td>
<td>{11,22}</td>
</tr>
</tbody>
</table>
plastic deformation. However, only four independent slip systems are available in titanium, and twinning is necessary in order to maintain the necessary deformation capability. The twinning planes in titanium are, \{1012\}, \{1121\}, \{1122\}, and \{1011\} depending on the temperature and deformation conditions [26,29]. It has been observed that twinning is the prevalent deformation mode at low strain in the layer adjacent to the substrate. The twinning plane observed in our study is of the \{1012\} type, one of the most frequent at ambient temperature. Multidirectional repetitive loading and increasing strain facilitate the initiation of different twin systems, leading to their intersection. Since the relative atomic movement is limited in deformation twinning, the gross deformation produced by twins is quite small and most of the plastic flow is due to movement of the dislocations [28,30]. It has therefore been reported that during the plastic deformation of titanium at ambient temperature, deformation twinning occurs first and accounts for less than 15% of the total plastic strain [31]. Our observations, which show that deformation twinning appears only in the layer in which the strain is low, are consistent with the literature.

As the strain increases, the further formation of twins will reduce the scale of the microstructure very rapidly. The dislocation activity will then predominate and the presence of a large number of twins and their intersection will hinder their movement. Hence, a high dislocation density occurs at twin boundaries in pure titanium [32]. As deformation twinning ceases to operate at moderate strain, an increasing dislocation density is observed which gives rise to the formation of lamellae shown in Figs. 4(a) and (b). Many dislocation walls are visible at lamellae boundaries (Fig. 4(c)) and microdiffraction patterns reveal that the local misorientation across them is small (Fig. 4(d)). It is worth mentioning that this microstructural feature is very similar to the intersecting of dense dislocations walls (DDWs) producing a parallelogram shaped structure during the initial stages of cold rolling of polycrystalline copper [33]. Our investigations clearly show the evidence of the transition from deformation by twinning to the formation of misaligned lamellae resulting from the dislocation activity but the exact mechanism would need some further clarification.

Besides, it has been shown that the deformation mode of pure titanium, twinning or slip, depends very much on the crystallographic texture of the material [34]. Therefore, the (0002) and \{1010\} pole figures of the \(\alpha\) phase were determined on a section perpendicular to the longitudinal axis of the bar which corresponds to the treated surface. Those pole figures reveal no preferred orientation which means that the relative activities of twinning and slip that have been observed do not result from the crystallographic texture.

4.2. Subdivision of lamellae and formation of a nanostructure

Close to the treated surface, i.e. at a depth of about 150 \(\mu\)m in specimens N3 and N4, the microstructure consists of narrow lamellae subdivided into dislocation cells or low angle disoriented blocks. These narrow lamellae look very much like microbands developed in deformed polycrystalline metals which formed by splitting of DDWs with increasing strain [33,35–37]. Microdiffraction patterns reveal that these microbands did not lie on any specific crystallographic plane.

At a certain level of strain, the dislocations are rearranged in order to minimize the total system energy. The dislocation cells observed inside the microband in Fig. 8 correspond to a typical configuration that can accommodate plastic deformation. It is suggested that these dislocation cells are at the origin of the formation of subgrain boundaries which will subsequently be converted into low angle disoriented blocks before becoming submicronic polygonal grains. This hypothesis is supported by Fig. 8 showing the presence of submicronic grains (marked by arrows) connected to the lamella and of the same size as the dislocation cells. It has also been observed that the dislocation density is often lower in submicronic grains than it is in lamellae (Fig. 5(c)). The microstructural observations indicate that additional strain accommodation is achieved by successive grain subdivision.

The formation of equiaxed nanograins was observed close to the top treated surface (Fig. 7(a)). It can be seen that the grain boundaries are well delineated and that a lot of nanograins exhibit a homogeneous contrast in dark field condition revealing the absence of a large deformation. This is confirmed by a high resolution TEM image of a nanograin showing no elastic distortion of the lattice (Fig. 9). The change in the ring patterns
also reveals a gradual increase in the rotation of the grain boundaries from low angle disoriented blocks to randomly orientated nanograins (Figs. 5(d), 6(b) and 7(b)). It is likely that nanograins result from the further evolution of submicronic grains due to the very high strain and strain rate on the top treated surface.

Owing to the structural characteristics of nanograins described above, it is assumed that a recrystallization process may play a role in their formation. That recrystallization can be considered as dynamic since it occurs during the SMAT process. Derby has carried out a systematic analysis of dynamic recrystallization in relation to two mechanisms: nucleation and the growth of recrystallized grains in a deformed material (classical recrystallization) and the formation of recrystallization by the gradual rotation of subgrains (rotation recrystallization) [38]. Both mechanisms lead to the break-up of the original grain structure. In classical or migration recrystallization, new grains are nucleated in areas of high plastic strain and grow into the deformed material. In rotation recrystallization, rotation of the cells and subgrains occurs gradually, until all the dislocations are absorbed by the grain boundaries.

According to the literature, nucleation and growth of recrystallized nuclei occur as a result of annealing after rolling or swaging of pure titanium at 400–600 °C [39,40]. Under severe plastic deformation conditions, such as torsional straining, the recrystallization temperature is reduced to about 300 °C [22]. A very high strain and strain rate are also produced during the SMAT process and a certain amount of adiabatic heating may occur but the exact temperature on the top treated surface is difficult to evaluate. Also, TEM examination does not show any evidence of nucleation and growth mechanism for nanograins but clearly indicate the importance of grain boundary rotation in microstructural development. It is therefore suggested that rotation recrystallization may play a major role in the final grain refinement mechanism during SMAT.

It is interesting to mention here that both α-titanium (hcp) and low stacking fault energy fcc polycrystals (including copper and stainless steel) are deformed by a combination of slip and twinning during grain refinement by SMAT. As the strain level increases, it can be observed in the case of stainless steel [13] and nickel-based alloy [41] that dislocation slip takes place first and twinning follows, which is the exact opposite of what we observed in our study of α-titanium. This corresponds to the results of studies conducted on α-titanium and low stacking fault energy fcc polycrystals during cold processing. It has been reported that, at a low strain level (<40%), fcc crystals are mainly deformed by slip and at a medium strain level (40–70%), by twinning and slip [39]. In contrast, the main deformation mode for α-titanium is initially deformation twinning up to a 20% reduction in the thickness of the rolling sheets and changes to slip after a 40% reduction [42]. It would seem that a high dislocation density and a reduction in the size of the microstructure are conducive to twinning in fcc metals while they are an inhibiting factor in α-titanium, which suggests that twinning dislocations experience greater resistance than glide dislocations when they cut through the forest in titanium.

It has been established that in most fcc metals, the twinning stress increases with the stacking fault energy [43], which means that twinning occurs more easily when the stacking fault energy is low, such as in copper (78 mJ/m²) or stainless steel (16.8 mJ/m²). However, it appears that this is not the case for α-titanium which has an hcp structure, because its stacking fault energy is very high (>300 mJ/m² [44]) and it deforms by twinning at low strain.

5. Summary

It has been shown in this study that a nanostructure can be produced on CP titanium using the SMAT process. Under our experimental conditions, the nanostructured layer on the top treated surface can be up to 50 μm thick. Optical and transmission electron microscopy investigations show the following changes in the initial coarse grain structure as the strain increases: (1) the onset of twins and the intersection of twin systems, (2) the formation of DDWs, (3) the nucleation of microbands associated with the splitting of DDWs, (4) the subdivision of microbands into low angle disoriented blocks and then highly disoriented polygonal submicronic grains, and (5) further breakdown of submicronic polygonal grains into randomly oriented nanograins.
The final grain refinement step to form nanograins has been discussed on the basis of a dynamic recrystallization process and there is microstructural evidence from our work that a rotation recrystallization mechanism may play a role.

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References