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Orthorhombic martensite formation upon aging in a Ti-30Nb-4Sn alloy

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HIGHLIGHTS

- A massive α″ martensite formation was observed after 24 h of heat treatment.
- Martensite formation occurs in the vicinity of α phase laths.
- Incorporation of Sn in the β phase reduces the strain needed to form α″ phase.

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ABSTRACT

The characteristics of orthorhombic martensite (α″) formed by step-quenching in a Ti-30Nb-4Sn (wt%) alloy have been investigated by transmission electron microscopy (TEM) and X-ray diffraction (XRD). According to literature, α″ lattice parameters depend mainly on the composition of the parent β phase. In this study, samples subjected to step quenching heat treatment presented α″ phase formation in the proximity of α phase laths, driven by two combined factors: solute rejection and lattice strain. Our results indicate that as the aging is prolonged, α″ becomes richer in solute content, which makes it more similar to the parent β phase. An average 2.55% lattice strain along [110]β directions was found to be necessary in order to obtain α″ from the β phase after 24 h of aging at 400 °C, followed by water-quenching. The initial lattice strain along the same direction was estimated at approximately 3.60% with zero aging time. The precipitation of the α phase does not inhibit a solute rich α″ phase formation.

1. Introduction

Titanium alloys have been extensively used in biomedical applications due to their biocompatibility, high specific strength, low elastic modulus and even superelasticity. These properties, however, depend a lot on microstructural features: the phases present, their morphology, volumetric fraction and behavior under strain [1,2]. As an example, recent studies have shown the promising use of Ti-Nb-Sn alloys in functionally-graded biomaterials, with both the elastic modulus and the tensile strength being optimized for a femoral hip stem prosthesis application [3,4].

Beta metastable titanium alloys display martensitic transformations from the β phase (bcc) to the α' (hcp) or α″ (orthorhombic) phases at a specific solute content range. Among Ti-Nb alloys, the α″ phase is formed after water-quenching from the β phase field in alloys with an Nb content higher than 17.5 wt% [5] and less than 36.2 wt% [1]. The β phase can also be transformed into α″ at room temperature as a result of a stress induced martensitic (SIM) transformation [6], and thus the α″ phase can be reversed into a β phase after heating above the martensite start temperature (Ms), leading to an observable shape memory effect [7]. Furthermore, a few experiments with High Energy X-ray Diffraction (HEXRD) reported the formation of an α″-like phase during continuous heating or isothermal heat treatments, foregoing the initial stages of α (hcp) phase nucleation [8,9]. This phenomenon was first observed by Duerig et al. [10] in Ti-10V-2Fe-3Al alloy, in which the isothermal α″ phase, presumably lean in Fe and V, has been formed at low temperatures (250–460 °C), favored by lower heating rates. At the time, the authors justified the lean α″ phase stabilization settled on the principle that the lattice strain needed to form α″ from the β phase is smaller than the strain needed to directly form the α phase, thus proposing the following phase transformation sequence during heating: β + α″ → β + lean

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Table 1
Composition of the experimental Ti-30Nb-4Sn (wt%) alloy.

Alloy	Ti	Nb	Sn	O	N
Ti-30Nb-4Sn	63.9 ± 0.3	31.8 ± 0.2	4.2 ± 0.1	0.131 ± 0.002	0.009 ± 0.001

Table 2
Crystallographic information among the phases studied.

CIF #	Composition	Phase	System	a' (nm)	b' (nm)	c' (nm)
44391 [16]	Pure Ti	β	ccc (229)	3.311		
43416 [17]	Pure Ti	α	hcp (194)	2.951		4.684
105248 [18]	Ti-20Nb at.%	α″	ortho (63)	3.166	4.854	4.652

Table 3
Modification of the β and α″ phase lattice constants with a solute addition.

Solute content	Lattice modification (10^{-3} nm)			
	a (β)	a' (α″)	b' (α″)	c' (α″)
1 at.% Nb [1]	0.013	1.364	−1.546	0.238
1 at.% Sn [2]	0.539	2.219	−2.369	0.153

$\alpha'' \rightarrow \beta + \text{lean } \alpha'' + \alpha \rightarrow \beta + \alpha$. The formation of α phase derived from the lean or isothermal α″ phase was recently elucidated by Barriobero-Vila et al. [11].

Several reports covered Ti-Nb based alloys in relation to the martensitic β/α″ transformation. According to Kim et al. [1], since there is a strict lattice correspondence between the β and α″ phases, the lattice transformation strain needed to form α″ from the β phase along a specific set of directions can be estimated using the lattice constants of each phase. The transformation strain needed is maximized when the loading axis is parallel to the [011]β directions. Furthermore, the transformation strain needed decreases with the increase of Nb, Sn and Zr contents as the martensite start (Ms) temperature declines [2]. Additionally, Bönisch et al. have demonstrated that the atomic rearrangement necessary to form α″ depends particularly on the parent β phase Nb content [12]. Liu et al. [13] identified clusters via HRTEM (high resolution transmission electron microscopy), which are rich in either Nb or Ti, proposing, in addition to the well-known α″ phase, the stress induced formation of another martensitic phase (δ martensite), also orthorhombic and apparently lean in Nb, in Ti-24Nb-4Zr-8Sn-0.1O (wt%) alloy. Most of these studies, however, were focused on the martensite formed from pure water quenching, or under stress/strain cycles [14], therefore none of them have properly

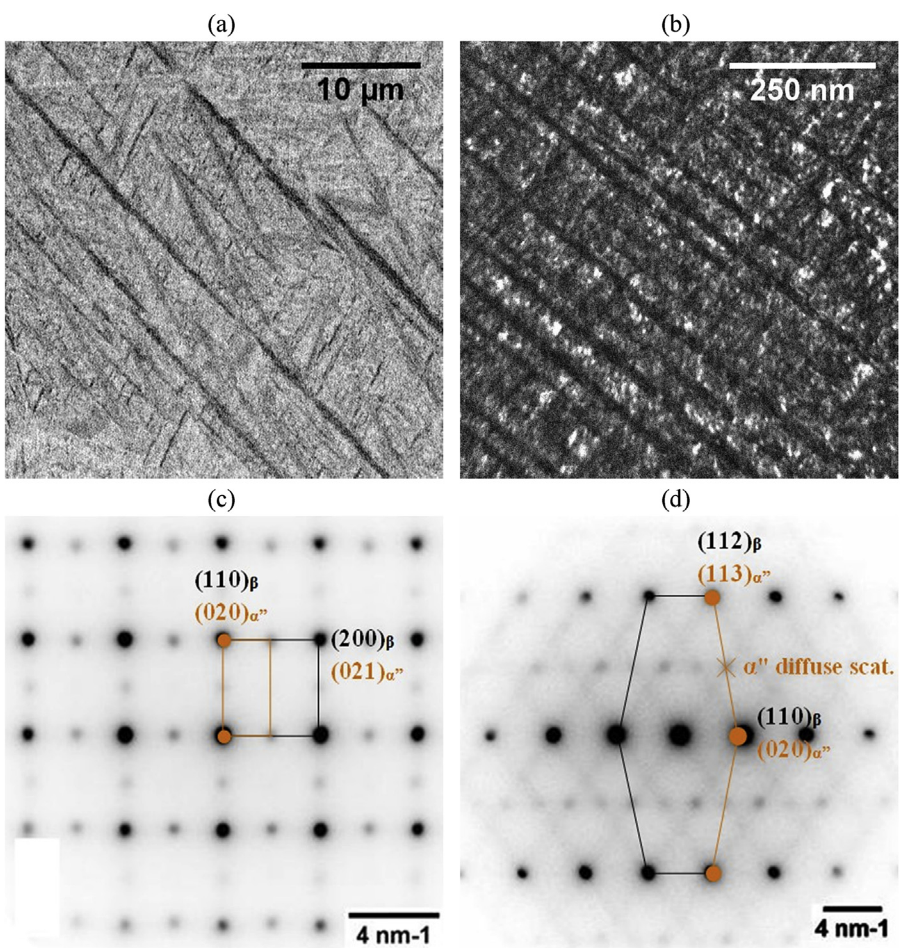

Fig. 1. Second phase formation on the WQ sample: FE-SEM BSE (a), Bright field TEM (b), SAD of ⟨100⟩β and ⟨113⟩β zone axes (c and d) and their respective key diagrams (Philips CM-200).