

Candidate Name: Abhishek Rastogi (2018MSZ8017)

Indian Institute of Technology, Delhi

Ph.D. Thesis Title: Stress-induced martensitic transformation role on microstructure-mechanical properties correlation in metastable β titanium alloys

Abstract

Stress-induced martensitic transformations (SIMT) in retained metastable phase are known deformation mechanisms that provide additional plasticity in several metallic alloy systems. Iron-, titanium- and high entropy-alloy systems exhibit metastable phases that show transformation plasticity due to twinning or martensite formation. Metastable β -Ti alloys, in specific, show significant potential for martensitic transformation through microstructure modification promoting leading to transformation-induced plasticity (TRIP). Typically, β (austenite) \rightarrow α'' (orthorhombic) and $\beta \rightarrow \alpha'$ (HCP) martensitic transformations are induced in them either by quenching or application of load. The two transformations differ from each other in terms of atomic shuffle displacement, with the former requiring less than the latter. As a result, $\beta \rightarrow \alpha''$ transformation can be considered as a crystallographically incomplete $\beta \rightarrow \alpha'$ transformation. Further, the influence of different alloying elements in metastable β -Ti alloys also plays an important role in driving the $\alpha'' \rightarrow \alpha'$ martensitic transformation. In addition, the microstructural state, in particular the β phase stability and β grain orientations, play an important role on the martensite transformation during deformation and eventual property enhancement in metastable β -Ti alloys.

This thesis work effectively demonstrates SIMT in metastable β Ti-10V-2Fe-3Al (Ti-1023) alloy resulting in enhanced cold deformability. The evidence of phase transformation from $\beta \rightarrow \alpha'' \rightarrow \alpha'$ martensites is observed in Ti-1023 alloy during continuous deformation. Microstructural characterization using electron backscattered diffraction (EBSD), X-ray

diffraction (XRD) and transmission electron microscope (TEM) analysis confirmed the transformation of α'' to α' . It is noted that the combined effect of lattice strain induced by cold rolling and the different alloying elements, drives the $\alpha'' \rightarrow \alpha'$ martensitic transformation. A correlation of the lattice strain and alloying elements on the transformation of $\beta \rightarrow \alpha'' \rightarrow \alpha'$ in Ti-1023 has been established. The observations thus confirm stress-induced martensite (SIM) been the prime reason for achieving enhanced cold deformability (up to $\sim 43\%$). Significant fraction of SIM provides preferred nucleation sites for new strain-free grains upon recrystallization annealing. The thermomechanical treatment results in grain refinement by $\sim 93\%$ in Ti-1023 alloys. Further, the influence of grain size on the SIM transformability based on micro hardness variations has been deduced. In addition, the role of the stability of β phase on the SIM transformation is elucidated using Electron Probe Micro Analyzer (EPMA) in the Ti-1023 microstructure. EPMA confirms the influence of diffusivities of alloying element (such as Fe, Mo, V, Cr and Al) as a function of recrystallization time on the β phase stability.

Furthermore, the surface characterization of micro indents using EBSD technique highlights that only a few grain orientations on the top surface showed the formation of stress-induced martensitic laths after micro indentation, suggesting that the SIMT is influenced by the grain orientations. Therefore, nanoindentation has been used to elucidate the grain orientation relationship on the SIM transformation behaviour during deformation and their eventual effect on the mechanical properties of Ti-1023. Austenite grains with specific crystallographic orientations of near $\langle 001 \rangle$, $\langle 101 \rangle$ and $\langle 111 \rangle$ are chosen. Nanoindentation is performed with two different diamond indenters viz. Berkovich and cono spherical. Nanoindentation reveals dominant stress-induced martensitic (of α'' type) transformation in the grains with near $\langle 111 \rangle$ crystallographic orientation, which contrasts with near $\langle 001 \rangle$ orientations showing only the presence of slip at the surface. In addition, lowest hardness (~ 4.1 GPa) is observed for near $\langle 111 \rangle$ grain orientations, while the hardness increased further

for the near $\langle 101 \rangle$ and $\langle 001 \rangle$ grain orientations (~ 4.3 GPa). The observations confirm that near $\langle 111 \rangle$ grain orientation is the most favoured grain orientation, followed by near $\langle 101 \rangle$ for enhanced SIM, while the near $\langle 001 \rangle$ grain orientation is the least favoured. Therefore, deformation micro-mechanisms across various length scales have been established by study of local deformation behaviour at the nanoscale.

Plastic deformation characteristics has been studied through different modes of localised deformation like micro and nanoindentation, however, the influence of SIMT on the overall material's mechanical behaviour is critical. Thus the influence of SIMT on the tensile deformation properties has been evaluated. The alloy show SIM transformation with strength enhancement, and the triggering stress variation with β grain size. However, the % strain to failure shows a significant decrease. Furthermore, the dual impact of SIM on the deformation and fracture behaviour is found, wherein the presence of SIM induces embrittlement during the plastic deformation, which resulted in absence of necking, despite enhancing the overall strength of the alloy by double yielding. Consequently, also providing indication for the absence of necking followed by non-uniform elongation during tensile deformation, which can be attributed to the embrittling actions of SIM. Since β grain size mainly governs the martensitic lath spacing and width, grain boundary engineering serves the foundation for creating high-performance Ti alloys.

Overall, the study highlights that the SIMT has successfully shown to result in achievement of the enhanced cold deformability. Furthermore, the high cold deformability is attributed to the orthorhombic- α'' martensites coupled with strain-induced $\alpha'' \rightarrow \alpha'$ martensitic transformations resulting in enhanced plasticity. SIM also acted as nucleating sites and eventually promoted grain refinement. Additionally, indentation studies have demonstrated the influence of grain orientations on the SIM transformability, providing confirmation that near $\langle 111 \rangle$ grain orientations are most favored, followed by near $\langle 101 \rangle$ for

enhanced SIM, while the near $\langle 001 \rangle$ grain orientations are the least favored. Finally, the dual impact of SIM on deformation and fracture behaviour is found, wherein, the presence of SIM induced embrittlement during the plastic deformation despite enhancing the overall ductility of the alloy by double yielding. Additionally, also provided indication for the absence of necking followed by non-uniform elongation during tensile deformation, which is ascribed to the embrittling actions of SIM. Consequently, the study evidently elucidates that β grain size and triggering of multiple variant martensites control the SIM transformability during plastic deformation. The study further demonstrates that effective control of SIM transformation potentially could provide strength-ductility enhancement via transformation-induced plasticity in metastable β titanium alloys.