ABSTRACT

Titanium and its alloys have garnered significant attention in various industries due to their exceptional properties, including high strength-to-weight ratio, corrosion resistance, and biocompatibility. As the demand for lightweight and durable materials continue to rise across diverse applications such as aerospace, automotive, biomedical, and sporting goods, titanium alloys have emerged as a preferred choice. Among the titanium alloys, Ti-6Al-4V, also known as Grade 5 titanium, is one of the most widely used titanium alloys due to its exceptional combination of properties, making it vital in various industries. However, the metastable β titanium alloys have gained significance due to their attractive properties like high biocompatibility, high fatigue, corrosion resistance, and young's modulus close to that of the human bones. Though β titanium alloys exhibit numerous advantages, certain limitations such as coarse β grains, limited formability, relatively higher density and susceptibility to phase instability, particularly at elevated temperatures or under load, limit its consideration among the titanium alloy classes. Despite this, ongoing research and advancements in alloy design, processing methodologies, including those involving severe plastic deformation (SPD) process, and understanding of phase stability aim to mitigate these challenges and thereby, expand the potential applications of metastable β titanium alloys in various industries.

Among the metastable β titanium alloys, β-21S Ti-alloy exhibit a unique balance of strength, toughness, and weldability, particularly suited for aerospace structures, marine components, and medical implants. β-21S Ti-alloy is used for manufacturing the tail plug for the B-777 engine. Most of the research on β-21S Ti-alloy has been carried out extensively on microstructural characterization and mechanical properties of various solution treatment and aging processes to improve the strength, but the ductility has significantly reduced. However, studies involving processing techniques such as SPD process to modify β-21S Ti-alloy microstructure and eventually effect on its mechanical properties and formability characteristics have not been attempted. Among the SPD processes, constrained groove pressing (CGP) is considered as the most suitable process for deforming materials in the sheet form. Hence, the broad aim of the current study is to achieve grain refinement using CGP process for an optimum combination of strength and formability in the β-21S Ti-alloy.

The β-21S Ti-alloy demonstrates good ductility with moderate strength in the solution treated (ST) condition. So, severe plastic deformation processes like constrained groove pressing (CGP) and constrained groove pressing – cross route (CGP-CR) techniques are used to achieve grain refinement in the β-21S Ti-alloy. The average grain size, tensile and anisotropy properties in the ST, CGP and CGP-CR conditions are determined. Aging of the CGP processed samples are carried out to alter the mechanical properties to achieve an optimum combination of strength and formability. The formability characteristics in terms of stretchability and drawability are characterized in ST, CGP and CGP-CR conditions. In addition, the tensile properties, anisotropy and formability in terms of stretchability, drawability and stretch flangeability of solution treated β-21S Ti-alloy has been compared with most widely used Ti-6Al-4V alloy to establish the specific advantages and limitations of β-21S Ti-alloy over Ti-6Al-4V alloy.

β-21S Ti-alloy sheets of 2 mm thick were used for the present study. Chemical composition analysis of the as-received material was conducted using X-ray fluorescence (XRF) and electron probe micro-analyzer (EPMA). Both methods predicted accurate quantification of elemental composition across multiple regions. Initially, the CGP and CGP-CR simulations were carried out using QForm software, considering sheet dimensions of 64 mm x 64 mm x 2 mm to estimate the effective plastic strain distribution and load required for CGP and CGP-CR processes. Grooved dies, flat dies, and constraint blocks were designed using SolidWorks software and manufactured using D2 steel via CNC machining. The Modified Hensel Spittel model proved to be the most suitable flow model for simulating the behavior of β-21S Ti-alloy sheets during CGP and CGP-CR processes. It was observed that strain inhomogeneity was more pronounced in the transverse direction (perpendicular to grooves) compared to the longitudinal direction (parallel to grooves) due to variations in shear and shear+bending regions. Experimentally, the β-21S Ti-alloy sheets were solutionized at 900°C for 30 min in uncoated and delta-glaze coated conditions. The uncoated sample exhibit a thick oxide layer while a glossy surface has formed on the delta-glaze coated sample. Oxygen diffusion during solution treatment is a significant concern for titanium alloys. Therefore, amount of oxygen diffused in the sample was evaluated through elemental mapping using electron probe micro-analyzer (EPMA). The investigations showed that the oxygen had diffused into the matrix of the uncoated sample while the delta-glaze coating acted as a barrier for the diffusion of oxygen atoms at higher temperatures. CGP experiments were conducted on a 300-ton hydraulic press for samples subjected to solution treatment using delta-glaze coating. Successful deformation was achieved in CGP up to a maximum strain of 3.48 (CGP-P3), while CGP-CR reached 4.64 (CGP-CR2). Simulation results has closely matched with the experimental values on the estimated force. The hardness inhomogeneity observed in experiments corresponded to the strain inhomogeneity predicted by simulations, as both values decreased with increasing strain. Intermediate annealing after CGP-P2 was attempted but it did not improve in reaching higher CGP passes, indicating the limitations of the CGP process compared to CGP-CR. Consequently, CGP-CR emerged as the superior process for achieving higher strains with lower inhomogeneity.

Microstructural analysis was performed using optical microscopy, X-ray diffraction (XRD), and field emission scanning electron microscopy (FE-SEM). Furthermore, electron backscatter diffraction (EBSD) and transmission electron microscopy (TEM) were utilized to analyze microstructural features and twin-like structures. The optical microstructure of samples in ST, CGP and CGP-CR conditions reveal the presence of twin-like structures alongside grain refinement, with higher magnification showing different morphologies and the coexistence of slip bands. With increase in the CGP passes, grain refinement of ~ 48% (CGP up to 3 passes) and ~62% (CGP-CR up to 2 passes) was achieved. In addition, the fraction of twin-like structures increased from 7% to 22%, suggesting that accumulated strains lead to stress concentration and nucleation of twins. XRD analysis confirmed the presence of a single β phase and suppression of stress-induced martensitic transformation (SIMT) during deformation. The peak shifts and broadening in XRD patterns indicated the strain-induced lattice distortion and grain refinement, with higher broadening observed in CGP-CR, signifying the potential for higher grain refinement. EBSD analysis further revealed the twin-like structures to be either $\{332\}\langle 113\rangle$ twins or kink bands and further grain boundary misorientation analysis suggests a transition from low angle grain boundaries to high angle grain boundaries with increasing strain. It was observed that during CGP-P1, the strain accumulated was primarily contributed to the higher grain refinement of approximately 39% along with the initiation of twin-like structures within the grains. However, at higher strains ($\varepsilon > 2.32$), microstructure evolution showed that primary twins had evolved from the kink bands, while the secondary twins have formed within the primary twins. Additionally, the peak intensity ratio of I(002)/I(112) increased with increasing strain accumulation, resulting in localized crystal rotation. The combined effect of the formation of kink bands and localized crystal rotation are expected to have played a vital role in activating the planes favorable for the deformation resulting in higher strain accumulation (ε=4.64) by CGP-CR process at room temperature. TEM and selected area electron diffracton (SAED) images confirm the presence of twins and kink bands, with increasing strain leading to the evolution of primary and secondary twins from kink bands. A hypothesis is proposed outlining the stage-wise evolution of complex microstructures during deformation, highlighting the role of dislocation density, strain accumulation, and crystal orientation changes in twin formation and grain refinement during severe plastic deformation processes like CGP and CGP-CR processes.

Tensile tests were carried out on samples in ST, CGP and CGP-CR conditions. In the case of CGP processed samples, the tensile samples were considered in both the longitudinal and the transverse directions, and the strain-hardening behavior was analyzed. The Hill's plastic strain ratio method was adopted to determine R-values. Hardness measurements were performed using a macro-Vickers hardness tester, and stretchability was evaluated using Erichsen cup test. The engineering stress-strain curves indicated that the ultimate tensile strength (UTS) increased with the number of passes for both CGP and CGP-CR samples, while elongation decreased. The presence of twins and kink bands played a crucial role in achieving an 18% increase in strength, with a 25% reduction in ductility, contrasting with other titanium alloys which typically experience a 10% strength increase but undergo a more significant 45-60% reduction in ductility after CGP-P1. Strain inhomogeneity significantly impact both strength and flow behavior, especially when samples were tested in the transverse and the longitudinal directions to the grooving. CGP processed samples tested longitudinally show higher strength compared to transverse samples, with enhanced hardening behavior in the latter. In CGP-CR samples, strength surpassed transverse CGP samples but remained lower than longitudinal ones. Despite variations in strength, similar elongation behaviors were observed, suggesting minimal influence of groove impressions on elongation. Flow softening behavior was observed in all conditions, with varying degrees of hardening and softening behavior in uniform plastic region. CGP samples exhibit improved hardening in the transverse direction than in the longitudinal direction which is attributed to microstructural heterogeneity. Finally, the room temperature mechanical properties and formability of β-21S Ti-alloy in solution treated condition and Ti-6Al-4V alloy in as-received condition are evaluated and compared. The alloys are subjected to various deformation modes, such as biaxial stretching, stretch flanging and deep drawing. Formability

was characterized in terms of forming limit curve (FLC), limiting dome height (LDH), hole expansion ratio (HER) and limiting draw ratio (LDR). *β*-21S Ti-alloy exhibit flow softening with similar strength, higher ductility and lower anisotropy than Ti-6Al-4V. Forming limit curves (FLCs) of both alloys were determined by performing tests in different strain paths using the simplified Hecker's method. The FLCs showed that limit strains of *β*-21S Ti-alloy are marginally higher than Ti-6Al-4V. In addition, the LDH, HER and LDR values of *β*-21S Ti-alloy are found to be higher than that of Ti-6Al-4V. Thus, the findings indicate better overall formability of metastable *β*-21S Ti-alloy making it more suitable than Ti-6Al-4V for room temperature forming applications.

Keywords: *β*-21S Ti-alloy, Ti-6Al-4V, constrained groove pressing, constrained groove pressing – cross route, twins, kink bands, slip bands, grain refinement, forming limit curve, limiting dome height, limiting dome height and limiting draw ratio.