

# Manufacturing of nanostructured Ti-6Al-4V alloy via closed-die isothermal multi-axial-temperature forging: microstructure and mechanical properties

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**Abstract** Manufacturing of nanostructured Ti-6Al-4V via a novel severe plastic deformation (SPD) method, named “multi-axial-temperature forging (MATF)” and study of its microstructure and some of its mechanical properties were the aims of this research. In this new technique, known multi-axial forging (MAF) method was developed by a multi-temperature procedure for producing nanostructure in rather brittle materials. Due to room-temperature workability limits and in order to prevent low-temperature embrittlement in Ti-6Al-4V, MATF process was started at elevated temperature of 725 °C, but in order to attain the smallest possible grain size, deformation temperature at subsequent passes was gradually decreased in steps of 75 °C. Grain refinement due to application of MATF was investigated using scanning electron microscopy (SEM). The results show that this process can severely refine the microstructure. The refinement occurred at a variable rate, so that it was significant at the first pass, but reduced substantially at subsequent passes. The average grain size of the final product obtained after the fourth pass was about 100 nm. The results of shear punch and hardness tests show that application of MATF process significantly increased the yield strength up to 50 %, ultimate strength up to 45 %, and hardness up to 26 % relative to those of the as-cast alloy, but it reduced its elongation slightly. The results of this research also show that MATF process facilitates production

of nanostructure in low-temperature brittle materials like Ti-6Al-4V alloy by means of SPD techniques.

**Keywords** Multi-axial-temperature forging · Ti-6Al-4V · Nanostructure · Shear punch test · Severe plastic deformation

## 1 Introduction

Processing by SPD refers to various experimental procedures of metal forming that may be used to impose very high strains on materials leading to exceptional grain refinement [1]. A unique feature of SPD is imposing high strain without any significant change in the overall dimensions of the work piece. In addition, the shape of the work piece is retained by using special tool geometries that prevent free flow of the material [1]. The main objective of a SPD process is to produce high strength parts from lightweight alloys that cannot be obtained through conventional thermo-mechanical processing [2]. Ultrafine-grained materials produced in this way have sub-micrometer grain structures and unique physical and mechanical properties and are generally designated as NANO-SPD materials [3]. Numerous techniques for SPD processing are now available. The major methods already established for fabrication of UFG materials are high-pressure torsion (HPT), twist extrusion (TE), multi-axial forging (MAF), and equal-channel angular pressing (ECAP) [3].

Multi-axial forging (MAF) is somehow a unique process in which the working is done at a temperature less than half of its melting point (i.e.,  $<0.5T_m$ ). Forging by MAF method can be performed in two ways; these are free and closed-die forging. Compared to free forging, closed-die method exhibits better dimensional precision, but it has received little attention [4]. In this technique, the sample is pressed in a channel die to a fixed strain, removed and rotated by 90°, then reinserted in the

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channel die and pressed to the same strain [4]. MAF process is widely used by researchers to produce UFG structure in materials such as AISI 1016 [5], Inconel 718 superalloy [6], magnesium matrix composite [7]. This method can refine microstructure into sub-microcrystalline (SMC) structure and consequently improves the mechanical properties such as strength and hardness. Another characteristic that usually develops in materials with SMC structure is low-temperature super plasticity. Low-temperature super plasticity in titanium alloys like Ti-6Al-4V is an important characteristic and can be used to produce structural components by superplastic forming (SPF) or isothermal forging at much lower temperatures (i.e., 600–700 °C) than conventional temperature range (i.e., 850–950 °C).

Nanostructured Ti-6Al-4V alloy via multi-axial forging method was developed in this research by a new multi-temperature procedure and the new technique named “multi-axial-temperature forging (MATF).” This technique allows one to obtain a UFG state in rather brittle materials. This process starts at elevated temperatures; therefore, in order to attain the smallest possible grain size, the deformation temperature should decrease gradually. The microstructure evolved was investigated by both optical and scanning electron microscopy and the mechanical properties of nanostructured Ti-Al-4V were investigated by standard shear punch and hardness tests.

## 2 Material and methods

Ti-6Al-4V ingot with chemical compositions shown in Table 1 was used in this work. Figure 1 shows the microstructure of the center of the as-cast ingot. This figure shows the initial ingot has a fine lamellar microstructure with a mean grain size of  $40 \pm 7 \mu\text{m}$ . In order to obtain the initial martensitic microstructure in all of the samples, all of the cast ingots were homogenized at 1100 °C for 20 min and then quenched in water. This heat treatment cycle is well known for titanium alloys before their deformation [8].

Samples with dimensions of  $21 \times 14 \times 14 \text{ mm}$  were multi-axially forged in a die in three orthogonal directions, as shown in Fig. 2. The initial height of the sample reduced though a sequence of pressure from 21 to 14 mm, so while the width of the specimen remained constant, its thickness strained by +0.41 normal strains. Therefore, the overall surfaces and the shape of the specimen remain constant. By ignoring the small amounts of shear strains, the effective strain in one pass of

MATF in plane strain condition was calculated from Eq. (1) to be 1.44.

$$\begin{aligned} \varepsilon_{eff}^{one\ pass} &= N \left\{ \frac{2}{9} \left[ (\varepsilon_{11})^2 + (\varepsilon_{22})^2 + (\varepsilon_{33})^2 \right] \right\}^{0.5} \\ &= N \frac{2\sqrt{3}}{3} \ln \frac{h_1}{h_0} = 3 \times 0.48 = 1.44 \end{aligned} \quad (1)$$

Where  $N$  is the number of pressing sequences and is equal to 3 for one pass of MAF. After each pressing sequence, the sample was removed, rotated, and reinserted into the die such that pressing can be carried out on the new height direction of the specimen. Figure 3 shows the dimensions and photograph of the die used in this research.

Due to workability limits of coarse grained alloy [9], the process was started at high temperature of 725 °C (Fig. 4), but in order to attain the smallest possible grain size at subsequent passes, deformation temperature was gradually decreased in steps of 75 °C. The final fourth pass was applied at 500 °C. Due to temperature instability of Ti-6Al-4V, all pressing tests carried out under isothermal condition with a strain rate of  $10^{-3} \text{S}^{-1}$ . For preparing isothermal condition, a ZWICK pressure testing machine was equipped with a resistance furnace having a temperature control panel with an accuracy of  $\pm 5 \text{ }^\circ\text{C}$ , superalloy anvils and cooling blocks and pipes, as shown in Fig. 4b. Since lubricating the interface between the specimen and the die is very important for achieving uniform deformation, a boron nitride solution which is a typical lubricant at elevated temperature deformation [9] was used in this research. The new multi-temperature MAF process carried out in this research (MATF) is shown schematically in Fig. 4a.

For microstructural observations, optical and scanning electron microscopy was used. The effect of grain refinement on mechanical properties was also studied by applying both standard shear punch and hardness tests. Worth mentioning that standard shear punch tests is an efficient method for producing strength data that can be well correlated to those found by the conventional tensile tests [10, 11]. Moreover, this method benefits from simple shape sample, easier preparation, and significant material saving (owing to the small sample size), as compared to those of the conventional tensile test.

Shear punch tests were carried out at room temperature on the specimens with thickness of 0.5 mm utilizing a ZWICK testing machine at a cross head speed of 0.5 mm/min. The 0.5 mm thick slices of the material sheared in a shear punch fixture having 3.18 mm diameter, flat cylindrical punch and 3.2 mm diameter receiving-hole. The applied load  $P$  was measured automatically as a function of punch displacement; the data were acquired by a

**Table 1** Mean chemical composition of Ti-6Al-4V alloy used in this study

Element	Ti	Al	V	C	Fe	Pb	Cu	Sb	Bi	Ni	Mn	Zn
Wt.	87.79	6.7	4.5	0.77	0.72	0.1	0.04	0.04	0.05	0.014	0.011	0.003