

Investigation and optimization of heat treatment process on tensile behaviour of Ti6Al4V alloy

Untersuchung und Optimierung der Wärmebehandlung auf das Dehnungsverhalten der Titanlegierung Ti6Al4V

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This article examines the microstructure and tensile strength of annealed Ti6Al4V alloy at different temperatures and times. Taguchi based L9 (3^2) design was used for the experimental design matrix and optimization of the tensile strength of heat treated samples. The optimum parameter combination was at the temperature of 950 °C and soaking time of 1 hour. ANOVA results show that temperature is the most influencing parameter and a regression equation of degree two was developed to predict tensile strength. Results of the microstructure show lamellar structure development within the samples heat-treated at 1000 °C.

Keywords: Annealing / Microstructure / Taguchi / Tensile strength / Ti6Al4V alloy

Schlüsselwörter: Anlassen / Gefüge / Taguchi / Zugfestigkeit / Ti6Al4V-Legierung

1 Introduction

Titanium is a widely used alloy in the chemical, aerospace and nuclear industries, due to its capability to withstand corrosion and high mechanical strength [1, 2]. Its application in the oil and gas industry can't be overemphasized, due to the requirement of tough and high strength materials in oil and gas exploitation. The high destructive failures in stainless steel and copper in the offshore environment has further necessitated the need for higher

strength materials. One of the specific areas of application of titanium in oil and gas industry is in its use for building high-pressure heat exchangers, a substantial space and weight without strength compromise have been achieved by high-pressure heat exchanger built by Rolls Laval using titanium [3]. However, the toughness of titanium might be one of its limitation in its applications in oil and gas field [2].

Materials strength and properties are defined by their microstructure. The desired strength of titanium

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can be achieved by heat treating the metal to achieve desired volume fraction, size of secondary α phase and morphology. Annealing, solution heat treatment and ageing are the major heat treatment techniques employed in microstructure alteration of titanium alloys [4]. The annealing process is known to be beneficial where a good fracture toughness is desired in which the basket weave-like Widmanstätten α microstructure is created [5, 6]. High cycle fatigue performance, improved erosive resistance have all been attributed to annealing process [7, 8]. Solution heat treatment and ageing are beneficial when it is done below the β transus temperature to obtain adequate ductility, wear resistance and hardness [9, 10]. Generally, mechanical properties of Ti6Al4V alloy have been observed to improve after annealing processes [11].

Ti6Al4V alloy is made of the $\alpha + \beta$ microstructure, the β transition temperature is germane to the heat treatment process and has been reported to be around 995 °C as shown in the equilibrium diagram of the Ti6Al4V alloy [12], *Figure 1*. Therefore, it is important to also know the material behaviour at different temperature during the transition of α/β phase.

The Taguchi design is a very powerful tool in the design of experiment, it employs the orthogonal array and the signal to noise ratio to reduce the variance of experiments and proves that quality is achieved through design and not inspection [13]. The method

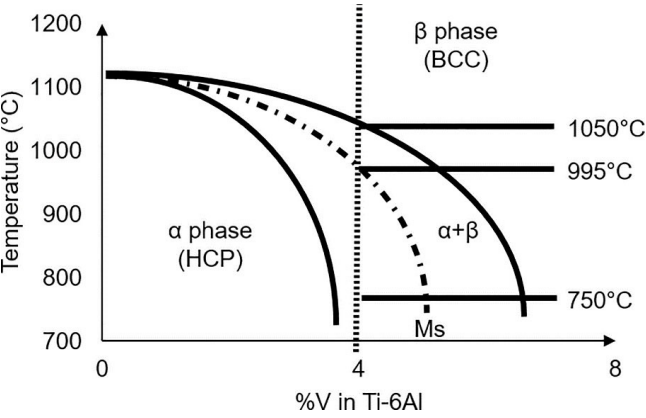


Figure 1. Ti6Al4V phase diagram.

Table 1. Chemical composition of Ti6Al4V alloy.

Element	Ti	Al	V	Fe	C	N	H	O	Others
Weight (%)	Remainder	6.10	4.0	0.15	0.03	0.018	0.002	0.13	Each < 0.10

Table 2. Experimental layout L9 (3^2) orthogonal arrays.

Trial no.	Heat treatment parameters		Test results
	A	B	
1.	1	1	
2.	1	2	
3.	1	3	
4.	2	1	
5.	2	2	
6.	2	3	
7.	3	1	
8.	3	2	
9.	3	3	

uses three standard signal to noise (S/N) ratios, which are larger the better, smaller the better and nominal the better. The selection of the S/N ratio depends on the process parameters to be optimized [14]. This method is applied in production industries and academics for product modification and optimization and this technique have been successfully applied in several industries [4, 13, 15, 16].

Therefore, this article investigates and optimize the process parameters of heat treatment to achieve an optimum tensile strength using the Taguchi method, the effect of annealing process on microstructural properties of Ti6Al4V alloy was also investigated.

2 Materials and method

The Ti6Al4V samples were as received 100 mm $\times$ 60 mm $\times$ 3 mm thick sheets with a tensile strength of 895 MPa, the chemical compositions of the materials are presented, *Table 1*. The samples were heat treated in a furnace then cooled in the furnace. The experimental matrix is based on the information from the technical data sheet and the 3 level design for the two factors (3^2) Taguchi design of experiment was adopted [17], *Table 2*. Factors

and levels and details of experimental approach are all presented, *Tables 3, 4*.

The samples were grinded using grinding papers from 320 grit size to 1200 grit size, they were then polished and etched using Kroll's Reagent. The microstructure was taken using the Olympus DP 25 optical microscope. The tensile samples were cut into ASTM E8 sizes, the test was carried out using the universal testing machine Zwick Roell 2250, three samples were used and the average value was reported [18]. The average tensile strength value and signal to noise ratio based on larger the better are calculated equations (1–2).

$$TS = \frac{\sum_{i=1}^n ts_i}{n} \text{ (MPa)} \quad (1)$$

$$SN = -10 \log \left(\frac{1}{n} \sum_{i=1}^n \frac{1}{ts_i^2} \right) \text{ (MPa)} \quad (2)$$

Table 3. Experimental design.

Level	Heat treatment parameters	
	A: Temperature (°C)	B: Time (min)
1	900	30
2	950	45
3	1000	60

Table 4. Experiment design of heat treatment and results.

Trial no.	Heat treatment parameters		Tensile strength (MPa)	S/N ratio
	A	B		
1.	900	30	861.92	58.71
2.	900	45	874.93	58.84
3.	900	60	944.83	59.51
4.	950	30	932.51	59.39
5.	950	45	920.19	59.28
6.	950	60	926.83	59.34
7.	1000	30	873.16	58.82
8.	1000	45	828.73	58.37
9.	1000	60	831.84	58.40

Where: *TS* is the tensile strength, *n* is the number of tests carried out, *SN* is the signal to noise ratio.

3 Results and discussion

3.1 Statistical modelling of annealing process parameters

Taguchi analysis method has been used to formulate the factors responsible for tensile strength results, the means effect plot of means of means and means effect plot for the S/N ratio and temperature is observed as the most significant contributing factor to the tensile strength of annealed samples and an optimum parameter of 950 °C and 1 hour have the largest S/N ratio, *Figures 2, 3, Table 5*. The interaction plot between tensile strength and soaking time at different temperature, *Figure 4*. The relationship can be said to have limited interaction with the parallelism among the lines representing the interactions [14].

The analysis of variance (ANOVA) results presents the contributions and significance of each parameter to the achievement of quality tensile strength, *Table 6*. Furthermore, regression analysis assists in predicting the changes among the variables (dependent and independent). The interaction of temperature (*A*²) has the highest contribution of 42.34 % in increasing the tensile strength of the annealed samples. The interaction between temper-

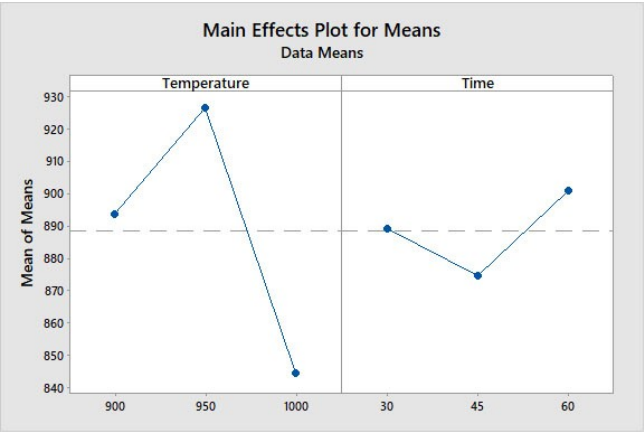


Figure 2. Main effect plot of means of means.

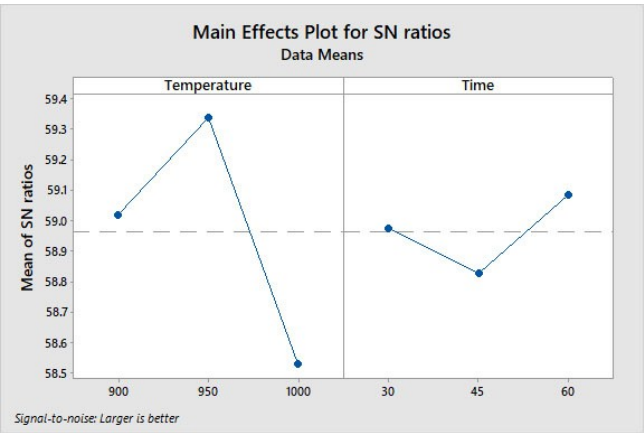


Figure 3. Main effect plot of means of signal to noise ratio.

Table 5. Response table for signal to noise ratios (larger is better).

Level	Heat treatment parameters	
	A: Temperature (°C)	B: Time (min)
1	59.02	58.97
2	59.34	58.83
3	58.53	59.08
Delta	0.81	0.25
Rank	1	2

ature and time ($A \times B$) exhibited the second highest contribution of 24.91 %. Temperature (A) with a contribution of 23.55 % is the third highest. Time (B) and interaction of time (B^2) have a percentage contribution of 1.39 % and 5.46 % respectively. These interactions shows that temperature is ger-

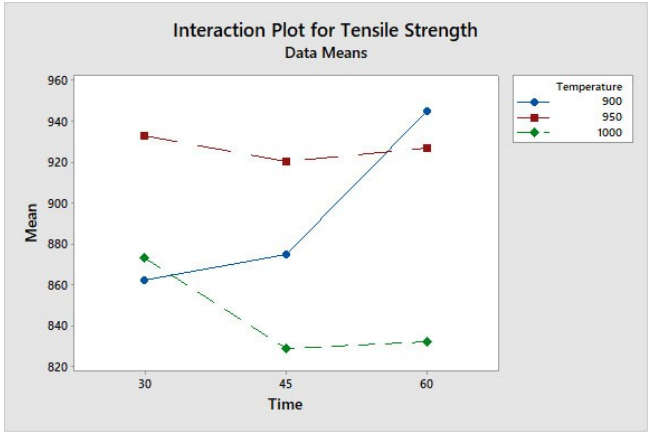


Figure 4. Interaction plot for tensile strength.

main to the success of achieving a good tensile strength. Furthermore, all parameters and their interactions are significant with P-Value less or equal to 0.05 at 95 % confidence level except the time interaction (B^2) with P-Value of 0.078, which is insignificant. The regression equation for predicting the tensile strength of annealed samples Equation (3), calculated using MINITAB-17 with R^2 value of 97.65 %.

$$\begin{aligned} \text{Tensile strength} = & -20897 + 44.90A \\ & + 31.51B - 0.02291A^2 + 0.0914B^2 \\ & - 0.04141AB \end{aligned} \quad (3)$$

3.2 Effect of heat treatment parameters on tensile strength of Ti6Al4V alloy

The tensile test carried out on heat treated samples showed that the samples failed in a ductile manner, Figure 5. A maximum tensile strength of 994.83 MPa was observed in the sample with an annealing temperature of 900 °C and soaking time of 1 hour, compared to the untreated material with a tensile strength of 895 MPa due to the increase in grain sizes in the microstructure which is known to improve the ductility of the sample [19]. The sample treated at an annealing temperature of 1000 °C and soaking time of 45 minutes had the lowest tensile strength of 828.73 MPa, the lower tensile strength could be attributed to the lamellar and α' martensitic structure observed in the microstructure

Table 6. ANOVA of annealing parameters.

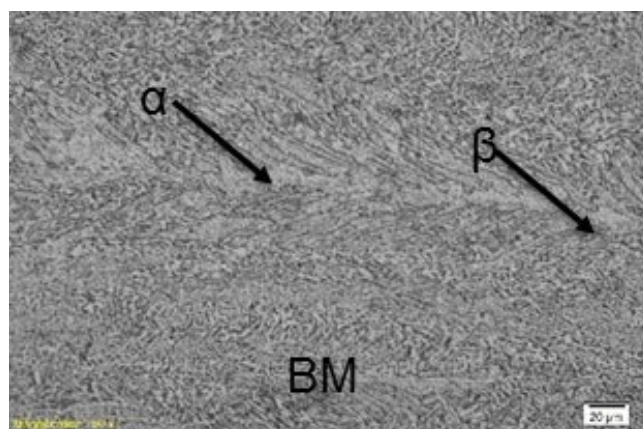
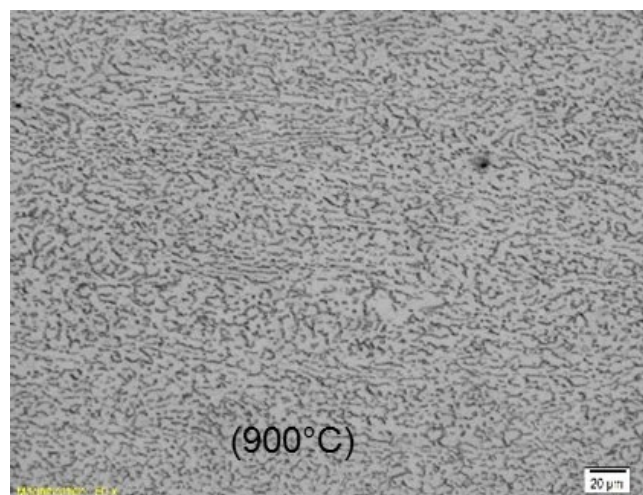
Source	DF	Seg SS	Contribution (%)	Adj SS	Adj MS	F-Value	P-Value	Remarks
Regression	5	15128.4	97.65	15128.4	3025.7	24.95	0.012	Significant
A	1	3648.6	23.55	6956.5	6956.5	57.35	0.005	Significant
B	1	214.9	1.39	2060.9	2060.9	16.99	0.026	Significant
A ²	1	6560.2	42.34	6560.2	6560.2	54.09	0.005	Significant
B ²	1	846.1	5.46	846.1	846.1	6.98	0.078	Insignificant
A×B	1	3858.5	24.91	3858.5	3858.5	31.81	0.011	Significant
Error	3	363.9	2.35	363.9	121.3			
Total	8	15492.3	100					

**Figure 5.** Tensile samples.

of samples heat treated within the 1000 °C temperature [20]. Samples heat treated within 950 °C, generally exhibited an improved tensile strength due to the even distribution of the α and β phases, compared with those heat treated within 900 °C and 1000 °C.

3.3 Effect of heat treatment parameters on the microstructure of Ti6Al4V alloy

The as-received metal and annealed metal at 900 °C, 950 °C and 1000 °C for 1 hour and furnace cooled respectively, *Figures 6–9*. The microstructure of the as-received material is made of the α and β phases, which expands as temperature increases, Figure 6. The annealing treatment at 900 °C and 950 °C shows a larger area fraction of

**Figure 6.** Micrograph of the base metal of the material.**Figure 7.** Typical micrograph of samples heat treated at 900 °C.

the α phase. At 1000 °C the primary α transformed into lamellar β due to increased temperature [21].

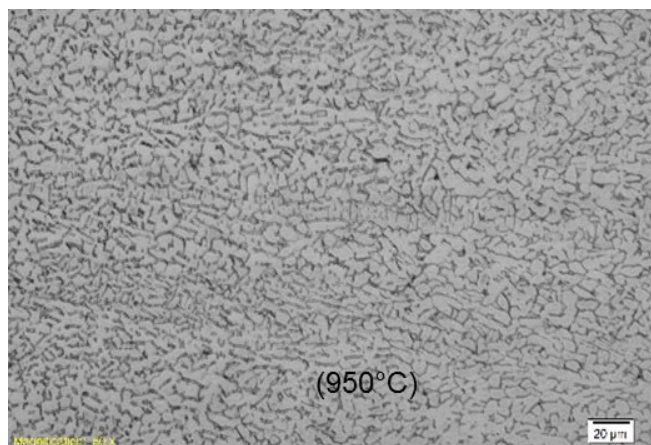


Figure 8. Typical micrograph of samples heat treated at 950 °C.

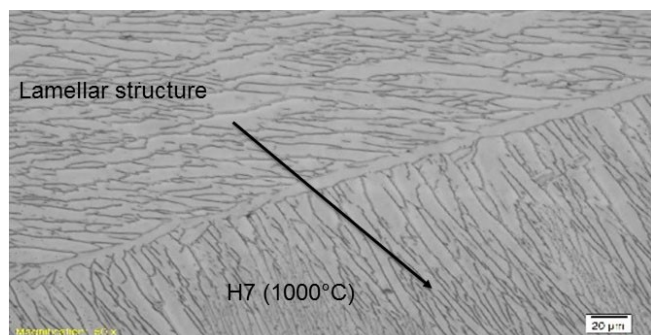


Figure 9. Typical micrograph of samples heat treated at 1000 °C.

4 Conclusions

The optimization and the investigation of the tensile and microstructural properties of annealed Ti6Al4V alloy have been carried out using the Taguchi method. The following conclusions are drawn from the experiment and optimization analysis:

1. Temperature plays the most important role in the improvement of the tensile strength of annealed Ti6Al4V alloy
2. The parameter combination of temperature of 950 °C and 1 hour soaking time is the optimum parameter for improved tensile strength.
3. The second-order mathematical model developed can be satisfactorily used in predicting the tensile strength of annealed Ti6Al4V alloy.
4. The microstructure of the annealed samples within 1000 °C shows lamellar structures and those within the 950 °C shows an improved ten-

sile strength due to the distribution of the α and β grains.

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