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Research Paper

FLOW BEHAVIOUR OF THERMAL CYCLED TITANIUM (Ti-6Al-4V) ALLOY

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Titanium is recognized for its strategic importance as a unique lightweight, high strength alloyed structurally efficient metal for critical, high-performance aircraft, such as jet engine and airframe components. Titanium (Ti-6Al-4V) alloy is an alpha beta alloy which is solution treated at a temperature of 950 °C to attain beta phase. This beta phase is maintained by quenching and subsequent aging to increase strength. Thermal cycling process was carried out for Ti-6Al-4V specimens using forced air cooling. The compression test was conducted for various temperatures at three different strain rates. Flow behaviour of different heat treated and thermal cycled Titanium (Ti-6Al-4V) alloy were investigated.

Keywords: Thermal cycling, Heat treatment, Solutionizing, Aging, Compressive strength

INTRODUCTION

Titanium is called as the “space age metal” and is recognized for its high strength-to-weight ratio. Today, titanium alloys are common, readily available engineered metals that compete directly with stainless and Specialty steels, copper alloys, nickel based alloys and composites. Titanium (Ti-6Al-4V) alloy is an alpha +beta alloy, which generally contains alpha and beta stabilizers and is heat treatable to various degrees. Alloying elements in titanium are classified as α stabilizers and β stabilizers. Alpha stabilizers such as oxygen and aluminium rise α to β transformation temperature. Beta stabilizers such as manganese, chromium, iron, molybdenum, vanadium, lower α to β transformation temperature and depending on the amount added may result in the retention of some â phase at room temperature. Alpha stabilizers (such as Al. only) are not heat treatable. Beta alloys are metastable and contain beta stabilizers (such as Vanadium) (Wood and Avor, 1972) are used to completely retain beta phase upon quenching. Ti-6Al-4V can be solution treated and aged to achieve significant

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increase in strength. Titanium alloys are needed to be heat treated to reduce residual stress developed during fabrication and to optimize special properties such as fracture toughness, fatigue strength and high temperature creep strength. The response of titanium and titanium alloys to heat treatment depends on the compositions of the metal and the effects of alloying elements on α-β Crystal transformation of titanium (Froes and Hajliberger, 1985). In addition, not all heat treating cycles are applicable to all titanium alloys because the various alloys are designed for different purpose. Ti-6Al-4v is designed for high strength at low to moderate temperatures. This alloy can be fully heat treated and can be used up to approximately 400 °C. Over 70% of all Titanium alloy grades melted on a sub grade of Ti-6Al-4V which is used in aerospace, air frame and engine components. Ti-6Al-4V alloys are heat treated for two different methods (Material Properties Handbook, 1994) in order to get optimum combination of ductility, machinability and structural stability by annealing and to increase the strength by solution treating. The heat treated specimens were subjected to thermal cycling up to 1500 cycles. Variations of True stress at different strain rates and temperatures for different thermal cycled specimens (500, 1000 and 1500 cycles) were analysed.

EXPERIMENTAL STUDY

The material used in this study is Ti-6Al-4v alloy. It has the following chemical composition (wt %). [Al-6.36, V-4.12, O-0.1562, C-0.015, Fe-0.04, N-0.0037, H-0.0045, Balance-Titanium]. Ti-6Al-4V specimens were machined (wire cut) to a dimension of 10 mm in length and 10 mm in diameter. Ti-6Al-4V specimens were subjected to two subsequent heat treatments. In the first heat treatment, the specimens are heated to a temperature of 750 °C for 4 hours and it is cooled in the furnace to reach the atmospheric temperature. In the second heat treatment the specimens are heated to a temperature of 975 °C for 1 hour and it is quenched in a solution containing water with 5% of caustic soda. After quenching (delay of 6 second), aging was done at 450 °C for 4 hours and then it is furnace cooled to reach atmospheric temperature.

The heat treated specimens are thermal cycled in a specially designed thermal cycling apparatus as shown in Figure 1. The basic thermal cycling apparatus comprises a muffle furnace, forced air cooling unit, electronic experimental controls unit and timer unit. The timer unit controls the heating and cooling time period of the cyclic process and regulates the flow of compressed air to the pneumatic cylinder through the electrically operated pneumatic valve. Further the experimental set-up as shown in Figure 2 consist of pneumatic components like double acting reciprocating air compressor, air control main valve, air filter, air regulator, air lubricator, electrically operated pneumatic valve and pneumatic cylinder. The pneumatic cylinder piston carries the cage, in which the test sample is placed. The cage is made up of stainless steel with three compartments to carry similar test samples in it. The timer unit counts the number of cycles and stops the process after completing the required number of cycles. Manual handling and monitoring of the sample during the thermal cycling process is minimized due to this automation. Pneumatic piston actuator was built to cycle the specimen in and out of the furnace. Where the required temperature was maintained in the furnace. The thermal cycle imposed had dwell time of 2 minutes in and out of the furnace. Samples were cooled by forced air cooling at a pressure of 2 bars. Heat treated specimens were subjected to thermal cycling in the range of 500, 1000 and 1500 cycles.

The compression test was conducted for various temperatures in the range of 200, 300 and 400 °C at three different strain rates in the range of 0.01, 0.1 and 1.00. Flow behaviour of different heat treated and thermal cycled Titanium (Ti-6Al-4V) alloy were investigated.

RESULTS AND DISCUSSION

Titanium (Ti-6Al-4V) alloys are heat treated (Solutionized and aged). The heat treated specimens were subjected to thermal cycling. The number of thermal cycle range is selected from 500 cycles to 1500 cycles in steps of 500 cycles. The compression test was conducted for various temperatures at three different strain rates. Flow behaviour of different heat treated and thermal cycled Titanium (Ti-6Al-4V) alloy were investigated.

Heat Treatments

Ti-6Al-4V specimens were subjected to two subsequent heat treatments. Primarily, the specimens are heated to a temperature of 750 °C for 4 hours and it is cooled in the furnace to reach the atmospheric temperature. Then the specimens are heated to a temperature of 975 °C for 1 hour and it is quenched in a solution containing water with 5% of caustic soda. After quenching (delay of 6 second), aging was done at 450 °C for 4 hours and then it is furnace cooled to reach atmospheric temperature.

Heating α + β alloy to solution treating temperature, produces a higher ratio of β phase. This partitioning of phases is maintained by quenching and subsequent aging, decomposition of the unstable β phase occurs, providing high strength. To obtain high strength with adequate ductility, it is necessary to solution treat at a temperature high in $\alpha - \beta$ field. Normally 25 to 85 °C (50 to 1500F) below the β transus of the alloy. If high fracture toughness or improved resistance to stress corrosion is required, â annealing or β solution treating may be desirable. However, heat treating $\alpha - \beta$ alloys in the β range causes a significant loss in ductility. These alloys are usually solution heat treated below the β transus to obtain an optimum balance of ductility, fracture toughness, creep and stress-rupture properties. If the β transus is exceeded, compressive properties of $\alpha - \beta$ alloys (especially ductility) are reduced and cannot be fully restored by subsequent thermal treatment (Srinivasan and Venugopal, 2008).

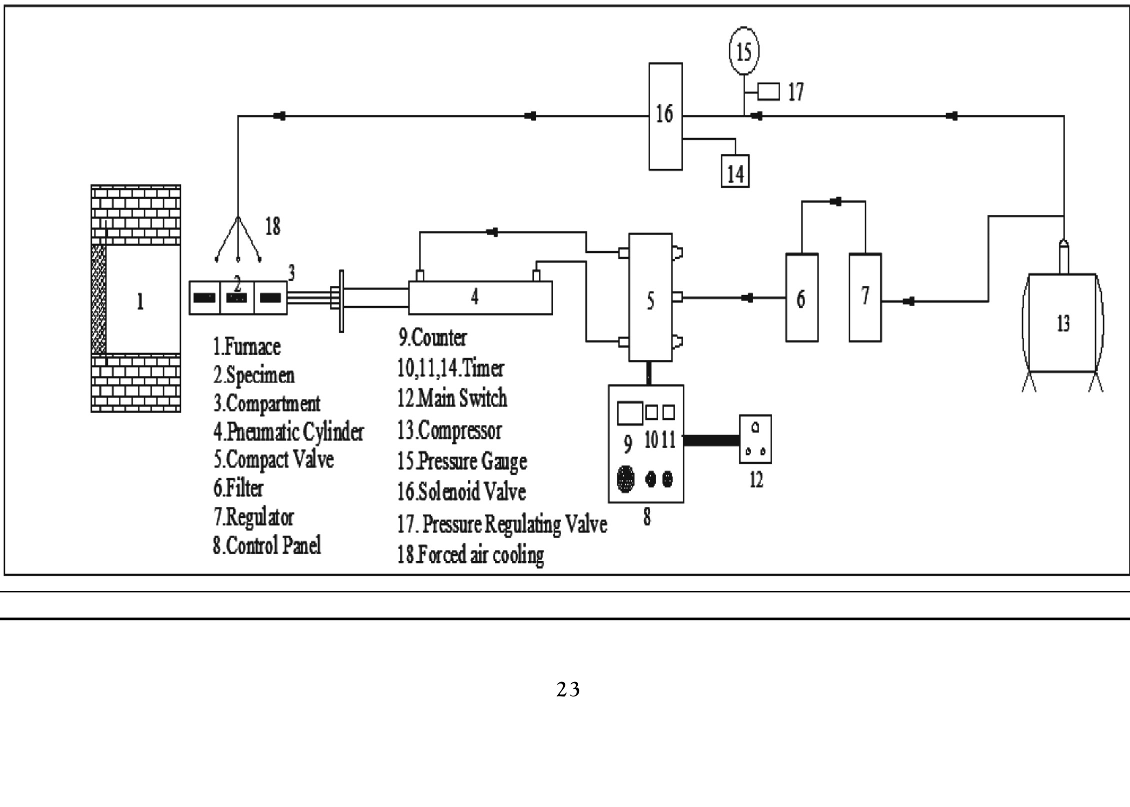
Thermal Cycling

The heat treated specimens are thermal cycled in a specially designed thermal cycling apparatus. The samples under the investigation are subjected to programmed cyclic heating and cooling temperatures in a muffle furnace for designed time period. Pneumatic piston actuator was built to cycle the specimen in and out of the furnace. Where the required temperature was maintained in the furnace. One heating and cooling operation of the sample is considered as one cycle. The samples are subjected to different number of cycles during the experiment. The thermal cycle imposed had dwell time of 2 minutes in and out of the furnace. Samples were cooled by forced air cooling at a pressure of 2 bars. Heat treated specimens were subjected to thermal cycling in the range of 500, 1000 and 1500 cycles. Manual handling of the sample in the thermal cycling process is very difficult, tedious process and not more accurate. Hence automation is necessary for material handling.

Thermal cycling is a temperature modulation process developed to improve the performance, strength and longevity of a variety of materials. During the thermal cycling process, materials are alternately cooled and heated until they experience molecular reorganization. This reorganization "tightens" or optimizes the particulate structure of the material throughout, relieving stresses, and making the metal denser and more uniform (thereby minimizing flaws or imperfections).

Figure 1: Experimental Set Up

Figure 2: Schematic Representation of Experimental Set Up



The tighter structure also enhances the energy conductivity and heat distribution characteristics of the material. The behaviour of the materials under thermal cycling conditions is affected by a number of factors such as the microstructure, thermal expansion coefficients of micro constituents, loading level and thermal conditions (Hongbin *et al.*, 1997; and Ismail and Kazim, 2004). Thermal Cycling minimizes “hotspots”, enhances cooling, and impedes the ability and tendency of metals to vibrate. Significantly reducing vibration as a factor in metal fatigue slows down the metal’s eventual failure or breakage (Xu *et al.*, 1994).

Hot Compression Test

Isothermal hot compression tests were conducted using FIE servo-controlled universal testing machine. The hot deformation of the material was investigated by conducting hot compression tests in the temperature range of 200-400 °C (in steps of 100 °C) and strain rate range of 0.01-1.0 s^{-1}.

Hot compression testing has become increasingly popular for several reasons, in particular: (a) uniform deformation can be maintained for large strains with proper lubrication, (b) the compressive state closely represents the conditions present in forging, extrusion and rolling process. Compression test on a cylindrical specimen is easy to obtain a constant true strain rate using an experimental decay of the actuator speed. It is convenient to measure the adiabatic temperature rise directly on the specimen and conduct the tests under isothermal conditions (Lin *et al.*, 2008). The size of the specimen was 10 mm in height and 10 mm in diameter. The load-stroke data were converted into true stress-true strain curves using standard equations. The flow stress values were corrected for the adiabatic temperature rise. The hot compression testing arrangement is shown in Figure 3.

Figure 3: Compression Testing Machine

Interpretations from Flow Curves

The flow curves are used to interpret the type of mechanism involved in deformation under particular condition. The flow curves for Heat treated and Thermal cycled Ti-6Al-4V alloy specimen with various temperatures (200,300 and 400 °C) and strain rate range of 0.01-1.0 S^{-1} are shown in Figures 4-12.

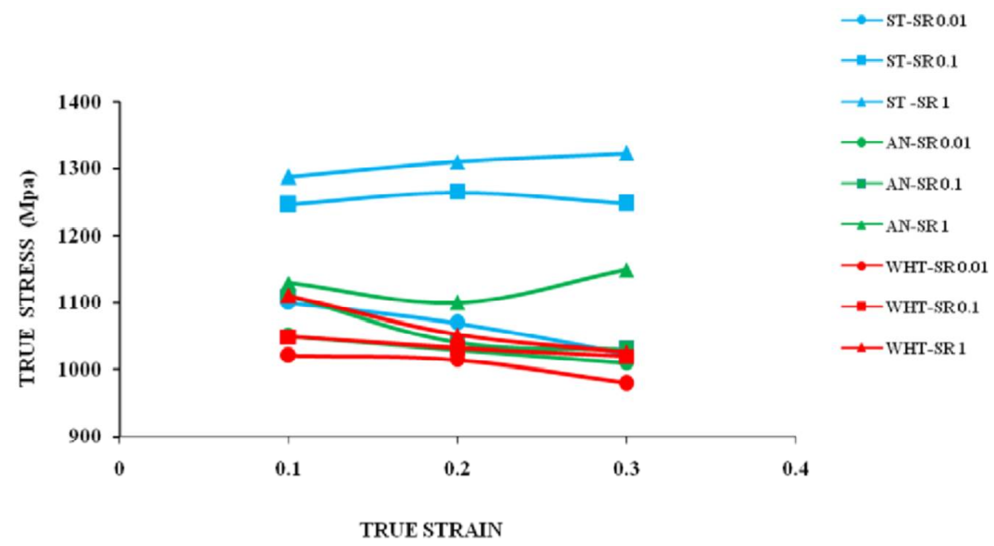
Flow curves of different heat treated and 500 thermal cycled Titanium alloy at different strain rates and at a constant temperature of 200 °C is shown in Figure 4. It is observed that, the flow stress decreases with the increase of temperature, but its variation with strain rate is low. At room temperature, the flow stress is found about 1240 MPa for specimens deformed at a strain rate of 1 S^{-1}. While at

400 °C, under the same strain rate level, it decreases to 740 MPa. The general features of the curves are similar to those at temperatures of 200 and 300 °C. It is also evident that the rate of work hardening at lower temperature in the initial stages of deformation is greater than that at 400 °C, and is slightly dependent on the strain rate (Lin *et al.*, 2008). When comparing these curves, the most obvious difference is the pronounced influence of the test temperature. As before, the flow stress obtained at 200 °C is greater when compared with those obtained for the other two higher temperatures. At a strain rate of 0.01 and at 200 °C temperature (Figure 4) the material exhibits a slight drop in flow stress indicating thermal softening behavior, However, for the rest of the temperatures, the stress strain curves show steady state flow without any peak stress. Figures 5 to 12 the stress strain behavior is different. For this strain rate, after‘ reaching the yield strength, the flow stress for all temperatures decreases with strain following different work hardening rates, except for 200 °C where steady-state low was observed. With regard to the specimens tested at a strain rate of 0.01 for a given temperature, the flow stresses decreased with the increase of strain, but a small rate of work hardening appeared when the temperature was greater than 300 °C. From the flow curves, it can be concluded that the work-hardening effect is pronounced at temperatures below 400 °C. Under the three tested strain rates and that the rate of work hardening decreases markedly with increase of temperature and strain rate.

From Figure 4, it is inferred that, true stress of the Solution Treated specimen (ST) increases upto 16% when compared to Without Heat Treated specimens (WHT). solution treated specimen has higher stress value than Annealed specimen (AN) at Strain Rate (SR) 0.1 and 1. True stress of the Annealed specimen increases upto 4% when compared to without heat treated specimens.

From Figure 5, it is inferred that, true stress of the solution treated specimen increases upto

Figure 4: Flow Curves of Different Heat Treated and 500 Thermal Cycled Titanium Alloy at Different Strain Rates and at a Constant Temperature of 200 °C



13% when compared to without heat treated specimens. solution treated specimen has higher stress value than Annealed specimen at strain rate 0.1 and 1. There is no substantial difference of true stress of the Annealed specimen when compared to without heat treated specimens.

From Figure 6, it is inferred that, true stress of the solution treated specimen increases upto11% when compared to without heat treated specimens. solution treated specimen has higher stress value than Annealed specimen at strain rate 0.1 and 1. Annealed specimen. exhibits higher stress when compared to without heat treated specimens.

From Figure 7, it is inferred that, true stress of the solution treated specimen increases upto 6.7% when compared to without heat treated specimens. solution treated specimen has higher stress value than Annealed specimen at strain rate 0.1 and 1. Annealed specimen.

Figure 5: Flow Curves of Different Heat Treated and 500 Thermal Cycled Titanium Alloy at Different Strain Rates and at a Constant Temperature of 300 °C

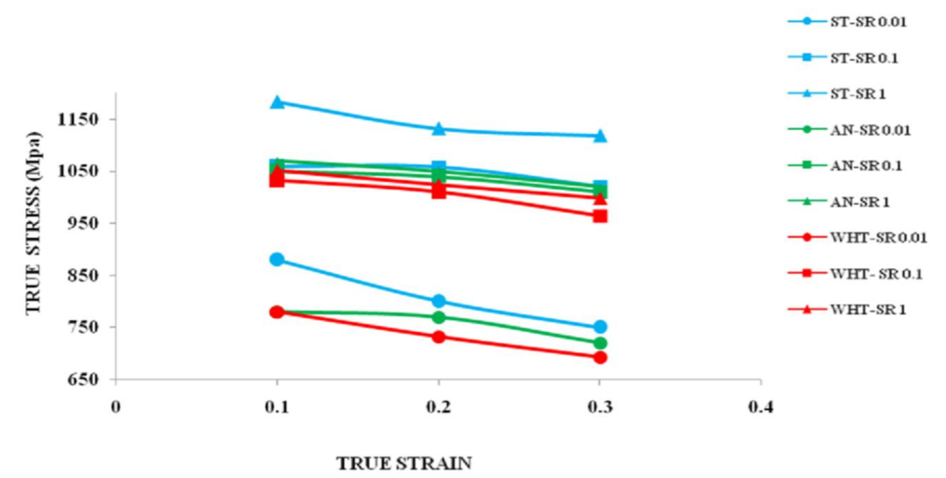


Figure 6: Flow Curves of Different Heat Treated and 500 Thermal Cycled Titanium Alloy at Different Strain Rates and at a Constant Temperature of 400 °C

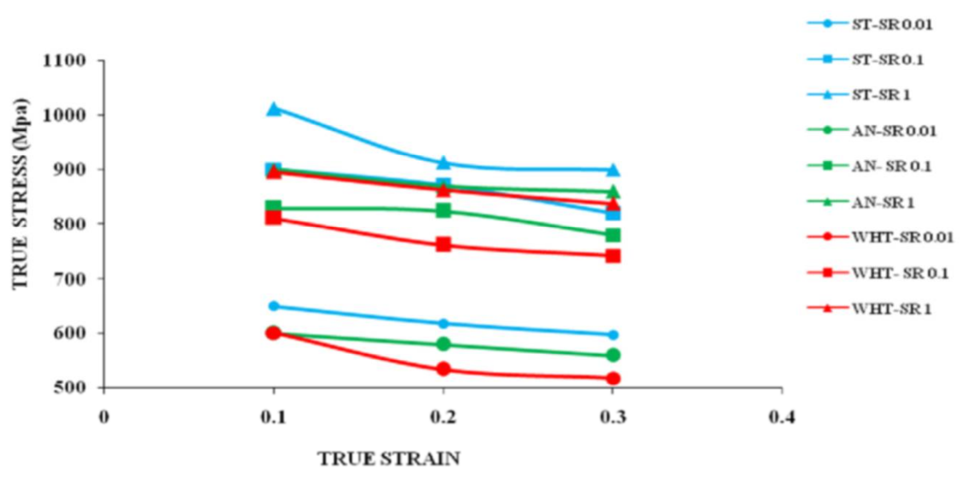
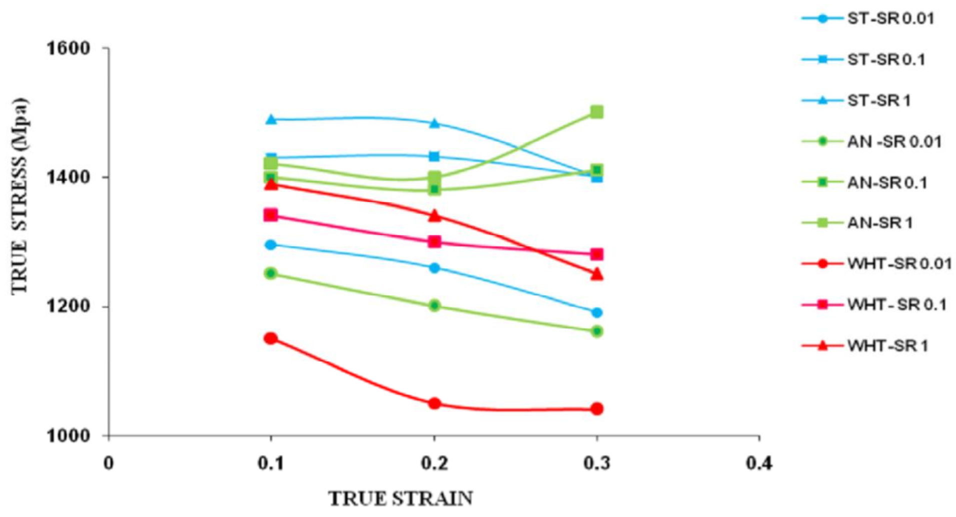


Figure 7: Flow Curves of Different Heat Treated and 1000 Thermal Cycled Titanium Alloy at Different Strain Rates and at a Constant Temperature of 200 °C



From Figure 8, it is inferred that,true stress of the solution treated specimen increases upto 5.8% when compared to without heat treated specimens. solution treated specimen has higher stress value than Annealed specimen at a strain rate of 0.1 and 1. Annealed specimens exhibits higher stress value when compared to Without heat treated specimens.

From Figure 9, it is inferred that, true stress of the solution treated specimen increases upto 5.5% when compared to without heat treated specimens. When compared to Annealed and without heat treated specimens, solution treated specimen has higher stress value at strain rate 0.1 and 1. Annealed specimen exhibits higher stress when compared to without heat treated specimens at strain rate of 0.1 and 1.

Figure 8: Flow Curves of Different Heat Treated and 1000 Thermal Cycled Titanium Alloy at Different Strain Rates and at a Constant Temperature of 300 °C

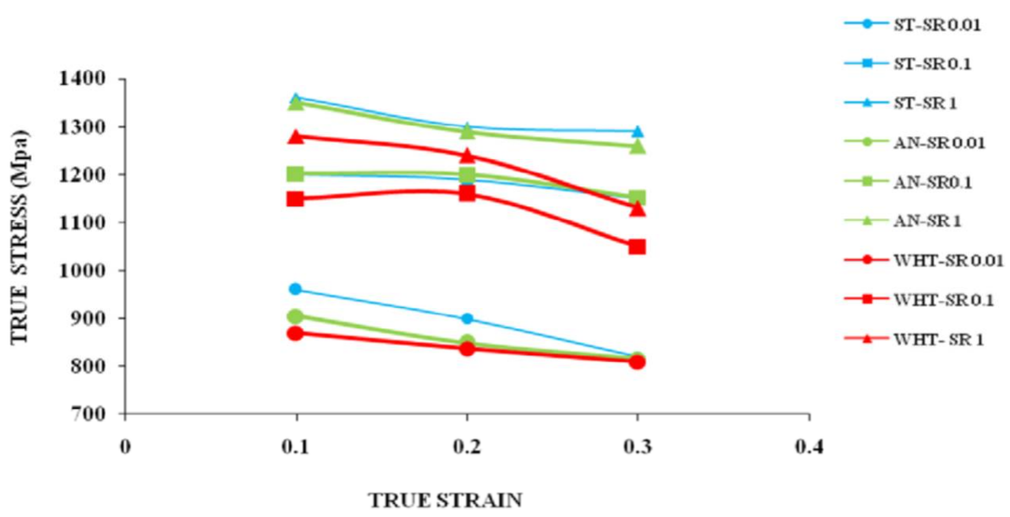
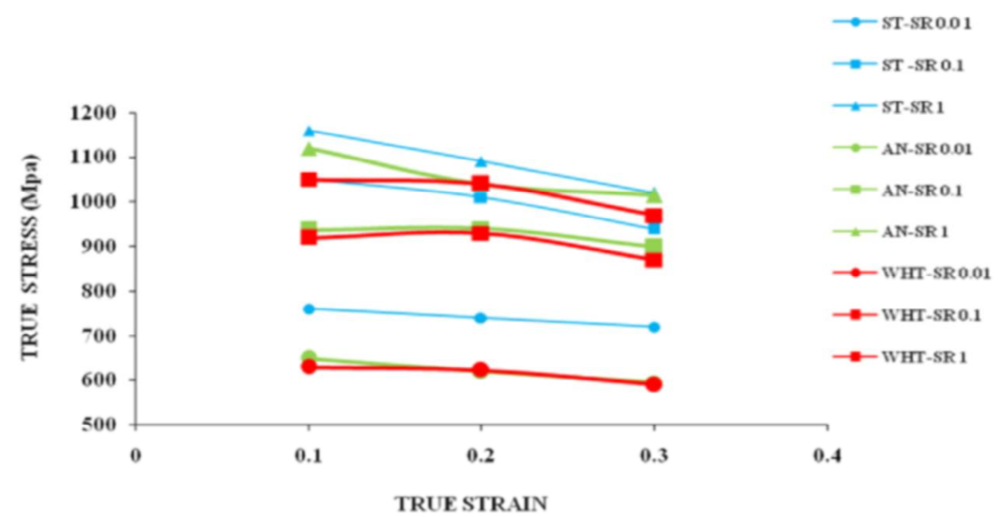


Figure 9: Flow Curves of Different Heat Treated and 1000 Thermal Cycled Titanium Alloy at Different Strain Rates and at a Constant Temperature of 400 °C



From Figure 10, it is inferred that, true stress of the solution treated specimen increases 6% when compared to without heat treated specimens. When compared to Annealed specimens, solution treated specimen has higher stress value than at strain rate 0.1 and 1. Annealed specimen exhibits higher stress when compared to without heat treated specimens at strain rate of 0.1 and 1.

From Figure 11, it is inferred that, true stress of the solution treated specimen increases upto 6% when compared to without heat treated specimens. Annealed specimen exhibits higher stress value when compared to without heat treated specimens at strain rate of 0.1 and 1.

From the Figure 12, it is inferred that true stress of the solution treated specimen

Figure 10: Flow Curves of Different Heat Treated and 1500 Thermal Cycled Titanium Alloy at Different Strain Rates and at a Constant Temperature of 200 °C

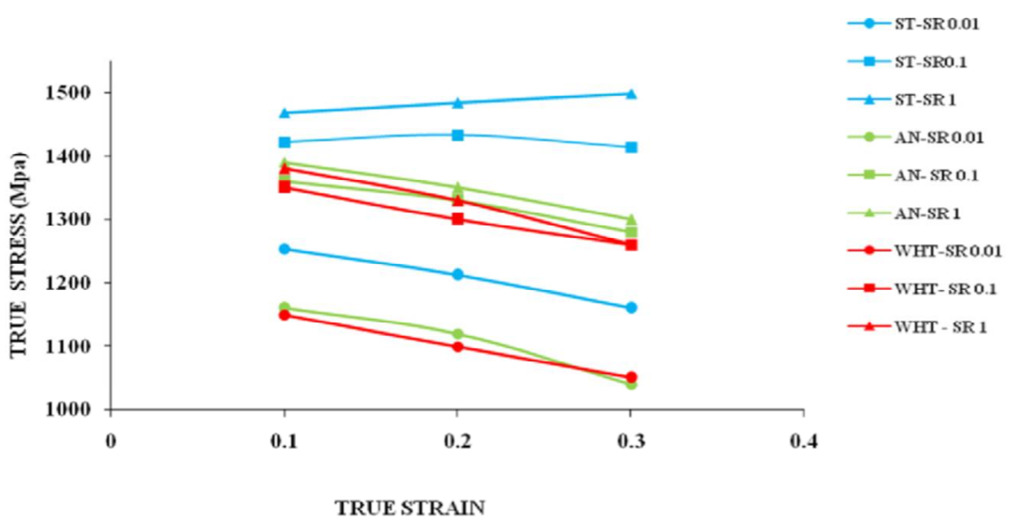


Figure 11: Flow Curves of Different Heat Treated and 1500 Thermal Cycled Titanium Alloy at Different Strain Rates and at a Constant Temperature of 300 °C

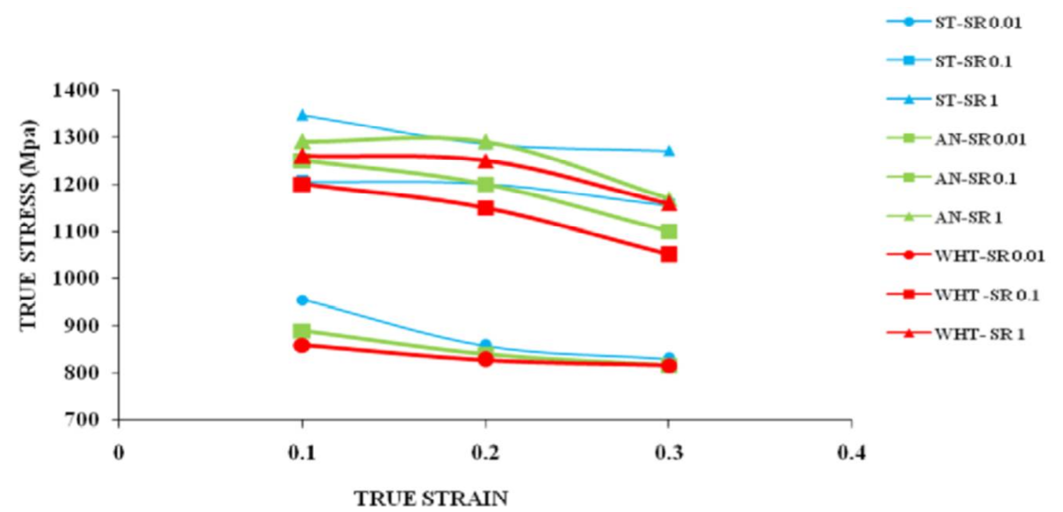
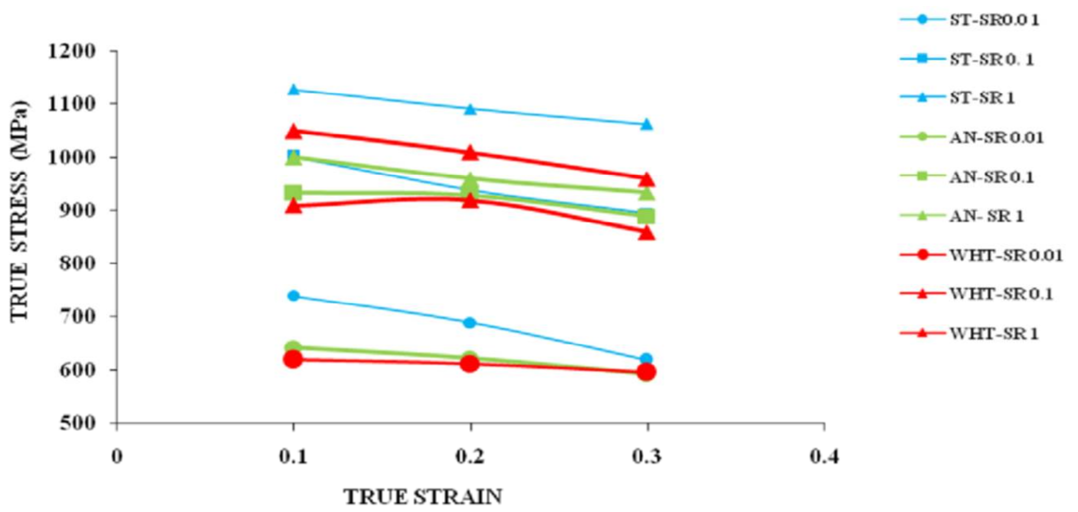


Figure 12: Flow Curves of Different Heat Treated and 1500 Thermal Cycled Titanium Alloy at Different Strain Rates and at a Constant Temperature of 400 °C



increases upto 6.3% when compared to without heat treated specimens. When compared to Annealed specimens, solution treated specimen has higher stress value. Annealed specimen exhibits higher stress value when compared to without heat treated specimens at strain rate of 0.1 and 1.

At lower temperatures, the strain hardening is more and steady state is obtained at higher temperature. At lower temperatures, the material exhibits severe work hardening followed by continuous flow softening. At higher temperatures, the flow softening behavior is distinctly different. The initial work hardening component is reduced and the material exhibits only steady-state flow behavior. This is good agreement with Wang *et al.* (2007). It is observed that peak stress increases with increasing of strain rates. But after peak stress, the rate of decrease in flow stress is high when the strain rate increases. The drop in flow stress after a peak at higher strain rate represents flow softening (Manish *et al.*, 2000). It can be found that flow stress decreases with decreasing strain rate. Also flow softening occurs at higher strain rates and steady-state flow occurs at lower strain rate. This is well agreed with Sivakesavam and Prasad (2003).

It is observed that compressive specimen after thermal cycling have good interface bonding and fine sub grains in the size of 0.2-0.5 μm. The refinement of grains greatly improves the strength of the mattering .Solution treated specimen increases the resisting forces of dislocation movement and also increases the difficulty of grain boundary sliding. Solution treated specimen attains greater strength and the hardness also increases for higher number of cycles. There is no effect of thermal cycling after 1000 cycles. True stress of the solution treated specimens shows higher value than Annealed and without heat treated specimens at 400 °C. True stress of the is high at 500 cycles when compared to without heat treated specimen and than Annealed specimens.True stress decreases upto 12% when the strain rate increases.

Vickers Hardness

Vickers hardness for Without Heat treated, annealed and solution treated specimens are shown in Table 1.

Table 1: Vickers Hardness for Without Heat Treated, Annealed and Solution Treated Specimens

Number of Cycles	Vickers Hardness of Without Heat Treated Specimens (WHT)	Vickers Hardness of Annealed Specimen (AN)	Vickers Hardness of Solution Treated Specimens (ST)
250	350	360	398
500	356	368	400
750	360	390	408
1000	360	398	413
1250	358	393	396
1500	355	383	388

It is observed that the Vickers hardness for Without Heat Treated specimens (WHT), Annealed Specimens (AN) and Solution Treated specimens (ST) are initially Increases with increase of number of thermal cycles up to 1000 thermal cycles and then it starts decreasing.

Vickers hardness for Solution Treated specimens (ST) found more than Without Heat Treated and annealed specimens. A recent study by Sharmilee *et al.* (2008) confirmed that an increase in micro hardness with an increasing number of thermal cycles, indicating that strain hardening is caused by dislocation generation and movement. In addition, repeated quenching from a high temperature of range 200 to 400 °C is expected to add to the concentration of vacancies, which form prismatic dislocation loops and increase the hardness as a result. The decrease in micro hardness with increasing number of thermal cycles is suggestive of softening by damage accumulation. The observations of decrease in micro hardness with increasing number of thermal cycles and damage accumulation are similar to those reported by Nikhilesh and Krishan (2006).

CONCLUSION

Heat treated (annealed and solution treated) and thermal cycled titanium alloy specimens (500-1500 cycle at 450 °C and 2 minutes dwell time) were subjected to Hot compression tests for different strain rate (0.01, 0.1, 1) and at different temperature (200 °C, 300 °C, 400 °C) by using a Universal testing machine. The flow behaviour of different heat treated and thermal cycled specimens were studied. Comparative study of flow behaviour of titanium alloy was made. The optimum Thermal Cycling was identified.

The following conclusions were made from the present study.

- The flow stress decreases with decreasing strain rate. Also flow softening occurs at higher strain rate and steady-state flow occurs at lower strain rate. At lower temperatures, the strain hardening is found to be high and it decreases with increase in temperature.
- The temperature and strain rate significantly affect the flow stress in the isothermal deformation.
- The flow stress decreases with increasing of deformation temperature and decreasing of strain rate.
- The flow stress of solution treated specimen showed higher stress value than without heat treated and annealed alloy over the ranges of temperatures and strain rates due to grain refinement and strengthening.
- The flow stress increases upto 500 cycles and then decreases in all heat treated specimens.
- The strength of the annealed and solution treated specimens are increases with number of cycles increases.
- The hardness increases at greater level up to 500 cycles and marginally rises from 1000 to 1500 cycles. When compared with, without heat treated specimen the percentage of hardness increases up to 9.5% for annealed and 12.8% for solution treated specimen.
- Solution treated and 1000 cycled Titanium (Ti-Al-4V) alloy was found to have more strength.

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