On the microstructural behavior of titanium alloy during non-optimal regime of superplastic deformation

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Abstract. In industrial superplastic forming, maintenance of the narrow ranges of condition required for optimal superplastic flow from beginning to end is a very difficult and challenging task. When these conditions required for optimal superplastic flow lie beyond a certain limit during deformation process, non-optimal process of superplastic deformation is observed during which the microstructure of the material changes very significantly. As the microstructure changes, the stress required to deform the material changes accordingly and the response of the material becomes dependent on the history of loading in this regime of superplastic deformation. A near alpha titanium alloy has been used in this study to find out those parameters of microstructure, which are varying significantly during non-optimal regime of superplastic deformation. Superplasticity tests have been carried out on tensile specimens at 930°C with a constant strain rate of $1 \times 10^{-4}$ s$^{-1}$ and successfully stopped at two different percentages of elongation. Results indicated that percentage of alpha phase, number of alpha grains per unit area, size of alpha grain, parameter of non-uniaxiality of alpha grain and grain boundary area of alpha grain, etc. varied significantly during non-optimal regime of superplastic deformation.

1. Introduction

Modern day technology demands materials which must have low density, high specific strength, excellent corrosion resistance and good biocompatibility. In this respect, titanium alloys are most favorable candidate materials and are being widely used for many industrial applications such as aerospace, automotive, biomedical etc [1-2]. Unfortunately, these titanium alloys are very hard and brittle in nature. Most of the above mentioned industries have already taken advantages to produce complex shaped components out of these materials in a limited number of manufacturing steps using superplastic deformation [3]. It is now well established that superplasticity is generally achieved in almost all polycrystalline materials with a fine grained microstructure when deformation of these materials is carried out under narrow ranges of strain rate and temperature [4]. Even, the much known brittle materials i.e. titanium alloys exhibit large elongations without fracture or formation of necks at these special regimes of superplastic deformation. These optimal regimes of superplastic condition are found experimentally by many researchers which are almost empirical in nature. To achieve superplastic flow in titanium alloys, three basic requirements must be fulfilled [5]. First, the average grain size should be smaller than 10 micron and these grains should be equiaxed. Second, superplastic deformation requires a relatively high temperature typically at or above $0.5T_m$, where $T_m$ is the...
absolute melting temperature of the material in degree Kelvin. Finally, these alloys must be deformed within a controlled strain rate mainly in the range of $10^{-4}$ to $10^{-2}$ s$^{-1}$. Of late, most of the research efforts on study of superplastic behavior of titanium alloys are mainly focused to find out the optimal regime of deformation for each alloy system [3, 6]. If all the above mentioned conditions are satisfied adequately during the superplastic deformation, optimal superplastic flow will be established. This behavior is mainly described by strain rate sensitivity parameter (m), the value of which must be greater than ~ 0.3 [7-9]. Main feature of optimal superplastic deformation is the occurrence of intensive grain boundary sliding associated with different accommodation mechanisms. The accommodation mechanisms observed in optimal superplastic deformation are mainly grain boundary migration, recrystallization, grain rotation, diffusional mass transport and slip in grains, etc [10]. As a result, the microstructure of the materials doesn’t change actively during optimal superplastic flow and the flow stress is a function of strain, strain rate and the grain size of the material [11-12]. Based on various deformation mechanisms operating during superplastic forming, the constitutive equation as described by many researchers is as follow:

$$\sigma = f (\varepsilon, \frac{d\varepsilon}{dt}, T, d)$$  

Where $\sigma$ is the flow stress, $\varepsilon$ is the strain, $\frac{d\varepsilon}{dt}$ is the strain rate, $T$ is the temperature and $d$ is the average grain size of the material [12-13]. In real technological applications, maintenance of these narrow ranges of condition required for optimal superplastic flow from beginning to end is a very difficult and challenging task [14-15]. In superplastic forming industry, the deformation process is carried out not at optimal, but close to the boundary of optimal superplastic condition that is called near super plastic regimes for which microstructure of the material changes significantly during the deformation process [14-17]. As the microstructure changes, the stress required to deform the material changes accordingly and the response of the material becomes dependent on the history of loading in this regime of superplastic deformation [15-16]. For simulation and modeling of these industrial superplastic forming processes, it is very much essential to develop a constitutive equation which can also take into account the major changes in microstructure of materials that takes place under non-optimal process of superplastic deformation.

In 1986, Ghosh et al. reported that the distribution of grain size of materials can change due to either grain growth or grain refinement occurring during superplastic deformation [18]. In superplastic deformation, strain is known to produce grain growth when a polycrystalline material deforms predominantly by diffusion creep and grain refinement when the same deforms by dislocation creep. In 1996, Zhou et al. developed constitutive equations taking grain size in to account based on deformation mechanisms i.e. diffusional creep, grain boundary sliding, dislocation creep and grain growth to describe the superplastic behavior of Ti-6Al-4V Alloy [19]. In 2009, Bhattacharya et al. carried out superplasticity tests on Ti-6Al-4V alloy in non-optimal regime and reported that the average grain size of Ti-6Al-4V alloy is changed from its initial values during this regime of superplastic deformation [14, 20]. In 2012, Bhaskaran et al. developed a constitutive equation taking grain size distribution into account to describe the behavior of various titanium alloys (i.e. near alpha titanium alloy and VT-9 titanium alloy) during near superplastic / non-optimal regime of deformation [15]. They concluded that the constitutive equation should also include complex microstructural transformation other than changes in grain size occurring in the non-optimal regime of superplastic deformation in order to improve its robustness and accuracy. As an input to the development of the constitutive equation for this regime of superplastic deformation, it is very much important to find out the microstructural parameters other than average grain size which are varying during the deformation processes. To the best of author’s knowledge, no serious research efforts have been undertaken till date on this matter. In this present investigation, therefore, an attempt has been made to find out those parameters of microstructure which are varying significantly during non-optimal regime of superplastic deformation of titanium alloy.
2. Research methodology

Development of an appropriate constitutive equation is the core subject of mechanics of solids. The investigations in this area are becoming much more complex when mechanical properties are strongly dependent on the microstructure of materials which changes significantly during the process of deformation. Such type of dependence is observed in the non-optimal regime of superplastic deformation. The present study aims to investigate the microstructural behavior of titanium alloy when the conditions of experiments are close to the boundary of optimal superplastic condition i.e. non-optimal regime of superplastic deformation. To satisfy the above condition, therefore, the limitations of the conditions required for superplastic flow need to be violated in one or more than one parameters. Out of the three controllable parameters (i.e. grain size, temperature and strain rate), it is very much convenient to control the strain rate externally. We also have control on the initial microstructure of the materials. As a result, the average grain size of titanium alloy must be less than 10 micron in order to achieve superplastic deformation in these alloy system. Temperature is another parameter which is most difficult to be actively controlled in the experiments due to large thermal inertia of the system. Hence, the temperature has fixed value from the range of temperatures required for superplastic deformation. The strain rate is the only parameter which can be controlled externally during the course of experiment by controlling the velocity of cross head of the universal testing machine [11]. Therefore, the experiment need to be carried out at a constant strain rate of $1 \times 10^{-4}$ s$^{-1}$ (which is close to the boundary conditions required for optimal superplastic flow) and strictly adhering to a constant temperature of 930°C (fixed value from the range of temperatures required for superplastic deformation).

3. Material and methods

3.1. Superplasticity test in non-optimal regime

The superplasticity tests have been carried out on tensile specimens made from as received rods of near alpha titanium alloy. These rods have undergone the mill process of cold rolling and annealing and its chemical composition is given in Table 1.

| Element | Al | V  | Mo | Fe | Si | C  | Ti   |
|---------|----|----|----|----|----|----|------|
| Weight %| 8.57 | 1.83 | 0.14 | 0.2 | 0.06 | 0.08 | Balance |

The beta transus temperature of this alloy was found experimentally to be 1040°C. Cylindrical specimens with gauge length 15 mm and diameter 5 mm have been made out of 12.7 mm diameter rod and tested on a universal testing machine (Instron 8801) having an electric heating furnace. The measurement of temperature of the specimen was made by thermocouples placed in immediate proximity to the gauge part of the specimen. It has been observed that the difference in temperature indicated by thermocouples attached with the furnace wall and the same placed at the surface of the specimen is about ± 2°C. The specimens were tested with a constant strain rate of $1 \times 10^{-4}$ s$^{-1}$ at 930°C and successfully stopped at two different percentages of elongation i.e. 160 and 466 percentages respectively. The constant strain rate was maintained by programming the exponential law for the velocity of crosshead of the universal testing machine [11]. At the end of experiment, the specimen was water quenched immediately in order to preserve the microstructure. Metallographic samples were prepared from the central portions of the tensile specimen after quenching. Kroll’s chemical reagent is used in order to reveal the microstructure of deformed specimens of titanium alloy [21].

3.2 Material characterization

The microstructure of both as received and deformed titanium alloy was characterized through optical microscope. To know the various phases present in the deformed tensile specimens after quenching,
XRD analysis was carried out on representative metallographic samples in a XRD Instrument (X’pert PRO PANalytical make) using copper as target. Hardness values for both initial and deformed titanium alloy have been measured using a micro-Vickers hardness tester (Leitz make) with a load of 300 gram on metallographically prepared samples. Minimum five numbers of readings have been taken on each sample in order to get reasonable statistics of the measured hardness values.

### 3.3 Calculation of microstructural parameters

Various types of microstructure are found in titanium alloys such as Lamellar, Equiaxed and Bimodal depending upon the different combination of the alpha and beta phases [22-23]. Size, shape and arrangement of these two phases primarily control the microstructural features of this alloy system. The geometrical parameter of these two phases such as size of alpha grain, parameter of non-uniaxiality of alpha grain, relative length of alpha grain boundary, orientation of alpha grain, volume percentage of alpha phase, and size of transformed beta grain, etc. are used to describe the microstructure of titanium alloys [24-25]. In this present study, we calculated percentage of alpha phase, number of alpha grains per unit area, size of alpha grain, parameter of non-uniaxiality of alpha grain and grain boundary area of alpha grain in an optical microscope (Leica DMI 3000M) by taking each individual alpha grain into account.

Three metallographic photographs were taken at about 1000x magnification along axial as well as transverse direction of material before and after superplasticity test. The shape of the alpha grain is assumed to be elliptical in nature and the length of both the major and minor axis was measured [26]. Length of the major axis was reported to be the size of the alpha grain. Parameter of non-uniaxiality of alpha grain was calculated as the ratio of length of major axis to that of minor axis. In case of two dimensional figures, the perimeter of alpha grain is reported as its grain boundary area. The average value with respect to minimum of three metallograph is reported.

### 4. Results and discussion

#### 4.1 Microstructural features and XRD analysis

The microstructures of the as received rod of titanium alloy in longitudinal and transverse direction are shown in the Fig.1. and Fig 2. These micrographs show that the original material contains small elongated alpha grains which are uniformly distributed throughout the beta matrix. The grain size distribution of alpha phase of as received titanium alloy in longitudinal direction is shown in Fig.3.

![Microstructure of initial titanium alloy in longitudinal direction: (a) Optical micrograph and (b) SEM micrograph](image-url)
Fig. 2 Microstructure of initial titanium alloy in transverse direction: (a) Optical micrograph and (b) SEM micrograph

Fig. 3 Grain size distribution of alpha grain of initial titanium alloy in longitudinal direction

Fig. 4 XRD plot of deformed titanium alloy after 160% elongation
XRD analysis has been carried out on representative samples to determine the phases present in the deformed material and are shown in Fig.4. From the XRD results, it has been confirmed that the deformed materials contain alpha phases in a beta matrix. All the peaks are showing the presence of alpha phases; but only the peak corresponding to the 2θ value equal to 39° shows the presence of beta phases. These results are in conformity with the previously reported results of commercially pure titanium, Ti-6Al-4V and Ti-6Al-7Nb alloys [21]. Detailed microstructural parameters (with their standard deviation values) of the initial titanium alloy in both longitudinal and transverse direction are shown in Table 2 and Table 3 respectively.

Tab. 2 Detailed microstructural parameters of initial and deformed titanium alloy in longitudinal direction

| Microstructural parameters | Initial material | After 160% elongation | After 466% elongation |
|---------------------------|------------------|-----------------------|-----------------------|
| Percentage of alpha phase | 22.5 ± 0.5       | 48 ± 0.5              | 55 ± 0.5              |
| Number of alpha grains per unit area(100 µm X100µm) | 3962 ± 12 | 306 ± 9              | 130 ± 6              |
| Average size of alpha grain (µm) | 1.9 ± 0.3 | 12.43 ± 0.41 | 8.13 ± 0.38 |
| Parameter of Non-uniaxiality of alpha phase | 1.55 ± 0.22 | 1.39 ± 0.01 | 1.35 ± 0.04 |
| Average grain boundary area of alpha phase (µm) | 7.86 ± 0.68 | 38.31 ± 0.73 | 28.6 ± 0.49 |
| Micro Vickers hardness (VHN) | 305 ± 2       | 318 ± 2              | 350 ± 3              |

Tab. 3 Detailed microstructural parameters of initial and deformed titanium alloy in transverse direction

| Microstructural parameters | Initial material | After 160% elongation | After 466% elongation |
|---------------------------|------------------|-----------------------|-----------------------|
| Percentage of alpha phase | 20 ± 0.5         | 45 ± 0.5              | 47.5 ± 0.5            |
| Number of alpha grains per unit area (100 µm X100µm) | 2423 ± 9 | 270 ± 7              | 174 ± 7              |
| Average size of alpha grain (µm) | 1.72 ± 0.34 | 9.67 ± 0.39 | 6.63 ± 0.34 |
| Parameter of Non-uniaxiality of alpha phase | 1.41 ± 0.02 | 1.29 ± 0.02 | 1.26 ± 0.02 |
| Average grain boundary area of alpha phase (µm) | 6.29 ± 0.75 | 31.30 ± 0.71 | 24.16 ± 0.72 |
| Micro Vickers hardness (VHN) | 301±2        | 315 ± 1              | 342±2                |

After constant strain rate (1*10^{-4} s^{-1}) test, the elongation has been found to be 160 and 466%. The microstructure in longitudinal direction as well as in the transverse direction at the central portion of the tensile specimen tested at constant strain rate of 1*10^{-5} s^{-1} is shown in Fig.5 and Fig.6. After comparing the microstructure shown in Fig.1 and Fig.2 with that of Fig.5 and Fig.6, it is clearly observed that there is a significant change in microstructure of titanium alloy during deformation in non-optimal regime. Detailed microstructural parameters of the deformed materials for different percentages of elongation in both longitudinal and transverse direction after the constant strain rate (1*10^{-4} s^{-1}) test are shown in Table 2 and Table 3. Our results indicated that the microstructural parameters, those varying significantly during superplastic deformation of titanium alloy in non-optimal regimes are percentage of alpha phase, number of alpha grains per unit area, average size of
the alpha grain, parameter of non-uniaxiality of alpha grain, and the grain boundary area of alpha grain. It was clearly observed that all these microstructural parameters get changed from initial material to the deformed material in both longitudinal as well as transverse directions. It has been revealed that the alpha grains grow at the expense of the beta phases in both longitudinal and transverse direction during the process of deformation. The size of the alpha grain is found to be higher after 160% elongation as compared to the same after 466% elongation in both longitudinal and transverse directions.

Fig.5. Microstructure of deformed titanium alloy after 160% elongation: (a) longitudinal direction and (b) transverse direction

Fig.6. Microstructure of deformed titanium alloy after 466% elongation: (a) longitudinal direction and (b) transverse direction

4.2 True stress vs. True strain behavior
Typical True stress vs. True strain plot for constant strain rate (1*10^{-4} s^{-1}) test, carried out at 930°C for both 160% and 466% elongation is shown in the Fig.7. For 160% elongation, this plot clearly indicates the occurrence of strain hardening during superplastic deformation in which the requirement of true stress for further deformation is gradually increasing with increase in the extent of deformation. The requirement of continuously increasing true stress to carry out further deformation could possibly be due to grain growth occurring in this material during deformation [27-30]. This has been clearly observed by comparing the initial microstructure of titanium alloy (Fig.1 and Fig.2) with that of deformed one (Fig.5). For 466% elongation, this plot clearly indicates the occurrence of strain...
hardening and strain softening during superplastic deformation in which the requirement of true stress for further deformation is gradually increasing with increase in the extent of deformation up to a certain percentage of elongation (i.e. 352%) and after that the requirement of true stress for further deformation is gradually decreasing. The requirement of continuously decreasing true stress to carry out further deformation after reaching a certain percentage of elongation (i.e. 352%) could possibly be due to recrystallization occurring in this material during deformation [15].

Fig.7. True stress vs. True strain curve for the superplasticity test of titanium alloy at 930°C with a strain rate of 1*10^{-4} s^{-1}

4.3 Strain induced grain growth and recrystallization

To achieve large elongation in superplastic materials, it is very much essential to maintain a small and equiaxed grain structure during the whole range of superplastic deformation [4, 29-31]. The mechanisms of superplastic deformation of materials are now well documented [5, 10]. Among all the mechanisms, most important is the intensive grain boundary sliding which is mainly contributing for such large elongations without the formation of neck [4, 10, 31]. Though, the majority of experimental data suggests that grain boundary sliding plays the dominant role in the superplastic deformation process, but it is reported that some additional accommodation processes (either diffusion controlled or dislocation motion) must accompany the grain boundary sliding to prevent the formation of neck [32]. Since diffusional flow is strongly dependent on grain size and dislocation creep is independent of the same, small grains are generally deformed by diffusion creep and large grains are generally deformed by dislocation creep during superplastic deformation [18]. Wilkinson et al. reported that grains can increase in size by several microns for every unit of applied strain when they are deformed at very slow strain rate [18]. There are already many literatures which report that strain induced grain growth taking place at a very slow strain rate during superplastic deformation [31, 33]. Since the contribution of diffusional creep to superplastic deformation in small grain materials is more significant and grain growth is inevitable at slower strain rate, it is attributed that diffusional creep leads to strain induced grain growth as observed in our experiment up to 352% of elongation [18, 31, 33]. Bhaskaran et al. reported that all the grains grow until they reach a critical size and as soon as the
grain achieves that critical size, it undergoes refinement [15]. With the formation of dislocation walls, large grains after reaching the critical size tend to break into small ones or new grain nucleate and grow on the surface of an existing large grain [16]. As the contribution of dislocation creep to superplastic deformation in large grain materials is more significant, it is attributed that dislocation creep leads to strain induced recrystallization as observed in our experiment after 352% of elongation.

4.4 Deformation induced phase transformation

After superplastic deformation of titanium alloy in non-optimal regime, the percentage of alpha phase in both longitudinal and transverse directions of the deformed material has been found to be increased in comparison to the same present in the initial material. This change in microstructural parameter, especially transition from beta to alpha phase could be facilitated in the presence of strain. Xue et al. observed deformation induced phase transformation in polycrystalline zinc sulfide at a temperature range of 900°C to 1150°C. They attributed that the conversion of hexagonal closed pack structure to body centered cubic structure below the allotropic transformation temperature i.e. 1020°C as strain dependent [34]. The percentage of alpha phase in the initial material was 22.5% and 20% in longitudinal and transverse direction respectively. However, after deformation it was found that in both longitudinal and transverse directions, the percentage of alpha phase has been increased significantly in comparison to the same present in the initial material as shown in Table 2 and Table 3. As the beta transus temperature of this titanium alloy is 1040°C and the experiments have been carried out at much lower temperature i.e. 930°C, this type of phase transformation may possibly be attributed to deformation induced phase transformation, which has been reported in other alloy system as well [35-39]. It is also observed that with increase in percentage of elongation, the percentage of alpha phases in the deformed material increases.

4.5 Microstructural parameters

A detail of microstructural parameters of both as received and deformed titanium alloy is given in Table 2 and Table 3. Microstructure plays a very important role in determining the mechanical properties of the titanium alloy, because practically all the mechanical properties are structure sensitive [22]. After superplasticity test, the micro-Vickers hardness values have increased from as received material to deformed material. As hardness is a structure sensitive mechanical property, it clearly indicates that this increase in hardness is the effect of change in the microstructure during superplastic deformation of titanium alloy in non-optimal regime. From this preliminary investigation, it is clearly observed that the microstructural parameters varying significantly are the percentage of alpha phase, number of alpha grains per unit area, size of the alpha grain, parameter of non-uniaxiality of alpha grain and grain boundary area of alpha grain. Hence, all these microstructural parameters need to be incorporated in the constitutive equation enabling the same to describe the materials behavior during superplastic deformation in non-optimal regime.

5. Conclusions

- Significant changes in microstructure have been observed in both longitudinal and transverse direction during superplastic deformation of titanium alloy in non-optimal regime.
- After superplastic deformation of titanium alloy in non-optimal regime, the percentage of alpha phase in both longitudinal and transverse directions of the deformed material has been found to increase in comparison to the percentage of alpha phase in the initial material. This type of phase transformation below the beta transus temperature has been attributed to deformation induced phase transformation. Strain induced grain growth and recrystallization is also observed during superplastic deformation of titanium alloy in non-optimal regime.
- The microstructural parameters those varying significantly are percentage of alpha phase, number of alpha grains per unit area, size of the alpha grain, parameter of non-uniaxiality of alpha grain, and grain boundary area of alpha grain.
All these microstructural parameters other than average grain size need to be incorporated in the constitutive equation describing the superplastic behavior of materials in order to increase its robustness and accuracy.

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