Recrystallization of Commercially Pure Titanium During and Following Hot Deformation

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Commercially available titanium(Grade 2)samples were deformed at high temperatures using a Gleeble 3500 thermo-mechanical simulator. These tests were performed under single hit(hot deformation and quenching)and double hit(hot deformation, un-loading, hot deformation and quenching)processes. The flow stress curves and microstructures of samples were studied after a single or double hit deformation to evaluate dynamic and meta-dynamic recrystallization processes, respectively. The results showed a significant influence of deformation temperature and strain rate on materials behaviour during deformation. Also, an extensive softening on the material observed during double hit deformation tests. The degrees of softening affected by temperature, strain rate and un-loading time. The double hit deformation process showed a significant grain refinement in α phase.

Keywords: *Titanium(Ti), dynamic recrystallization, meta-dynamic recrystallization, grain refinement*

1. Introduction

Titanium alloys exhibit mechanical and physical properties that fit aerospace applications in which light weight, good wear and corrosion resistance are required. In addition, they provide significant economic benefit in these applications. However, these unique properties are strongly affected by the chemical composition, microstructure, deformation and heat treatment history. Commercially pure titanium(CP-Ti), although being the most the basic titanium alloy, is being used in many industries as a structural material due to its excellent formability at high temperature and. as a biomaterial due to its chemically inertness and biological compatibility.

CP-Ti attracted significant research interest because of its excellent response to hot-deformation and thermo-mechanical processing(TMP) compared to alloyed Ti. However, the hcp crystallographic structure of CP-Ti, results in poor deformability at low temperatures. This is due to the limited slip systems of hcp structures at room temperatures[1)] but the hcp structure still has complex deformation systems at high temperatures, compared to the fcc and/or bcc structures in steel. Unlike the numerous hot-deformation studies on steel structures(for instance[2-3)]), there is limited number of such studies on titanium alloys[4-6)]. Reed-hill *et al*[6)] have developed a constitutive model to relate the flow-stress of CP titanium to the temperature and strain rate. Nemat-Nasser *et al*[4)] have studied the deformation behaviour of CP titanium in a wide range of deformation temperature and strain rates. They found that the stress-strain curves of CP titanium show very different deformation patterns at different temperatures. In more recent work, Zeng et al.[5)] developed a deformation condition map for predicting the flow-stress of CP titanium under different deformation conditions. Notwithstanding these studies, many aspects of hot-deformation mechanisms and recrystallization in CP titanium are still unresolved and further investigations need to be done to fully understand the behaviour of CP and other titanium alloys during hot-deformation.

An important issue that has received little attention in the literature is the deformation characteristics of Ti alloys following hot-deformation. Many studies on steel structures, e. g.[3,7)], have shown that after hot-deformation and during slow cooling or annealing the deformed material at high temperatures, severe softening occurs due to static recrystallization(SRX) or meta-dynamic recrystallization(MDRX). It is expected that similar phenomena ought to occur in titanium alloys and it is therefore important to analyses the SRX and MDR phenomena in Ti alloys. Due to very high stacking fault energy and rapid self diffusion in the bcc phase, the recrystallization rate in β-titanium is limited[8)]. But α-Ti with a hcp structure should experience recrystallization during and after hot-deformation.

The present research is preliminary concerned with flow-stress behaviour of CP titanium under different conditions. The microstructural development during such hot-deformation processes has also been studied and in addition, the behaviour of deformed CP Ti alloys has been studied by using double-hit deformation tests.

2. Experimental Procedures

12. 7 mm diameter rods of commercially pure titanium alloy, Grade 2(0. 07% C, 0. 25% Fe, 0. 15% O, balance Ti) were used in this study. Samples for compression tests, 12. 7mm diameter and 19 mm in length were cut from the as-received rods. Hot-compression tests were carried out in a Gleeble 3500 thermo-mechanical simulator. Samples were heated to 1223 K at a rate of 2. 5 Ks^{-1} and held for 180 s, aiming to achieve a single phase β-microstructure. To study the deformation behaviour of this material, samples were cooled to

1123,1023 or 923 K at a cooling rate of 5 Ks^{-1}. Following cooling, samples were deformed at three strain rates of 1, 0. 1 and 0. 01s^{-1} followed by rapid water quenching. To study the behaviour of materials following deformation, double-hit deformation tests were performed. In this regard, after the first deformation pass, samples were unloaded for different times and then a second deformation was applied under the same strain rate as the first deformation. The samples were quenched after the second deformation so as to freeze the microstructure existing at high temperature.

The resulting flow curves were further analysed as shown below and microstructural development was studied using optical microscopy following standard metallographic practice.

3. Results and Discussions

3. 1 Flow Stress Curves

1) Single hit deformation

A number of stress-strain ($\sigma - \varepsilon$) curves derived from hot-deformation tests at different temperatures (T) and strain rates ($\dot{\varepsilon}$) are presented in Figure 1. As expected the flow curves are heavily dependent on the temperature and strain rate. After deformation at 1123 K and for all strain rates, the flow curves exhibited a finite peak stress followed by a long steady state condition, indicating the typical shape of dynamic recrystallization. In these flow curves the steady state continued until disruption of the test. However, the absence of a peak stress means that DRX is not the only softening process and dynamic recovery (DRV) should also be considered as a possible restoration mechanism. It is worth nothing that the typical shape of a flow curve that displays DRV as the dominant softening mechanism, is an increasing flow stress to a maximum value (steady state) followed by a prolonged steady state stress2).

The behaviour of samples deformed at 1023 K is strongly dependent on the strain rate. At a strain rate of 0. 01 s^{-1}, the flow stress curves show typical DRX behaviour with a small peak stress (Figure 1c). By increasing the strain rate, a steady-state flow stress is not observed and the stress increased continuously. The increasing rate of flow stress is amplified by an increase in the strain rate as shown in Figures 1b and c.

After deformation at a temperature of 923 K and at all strain rates, the flow stress curves show work hardening. In these curves, the flow stress increases continuously with increasing strain rate.

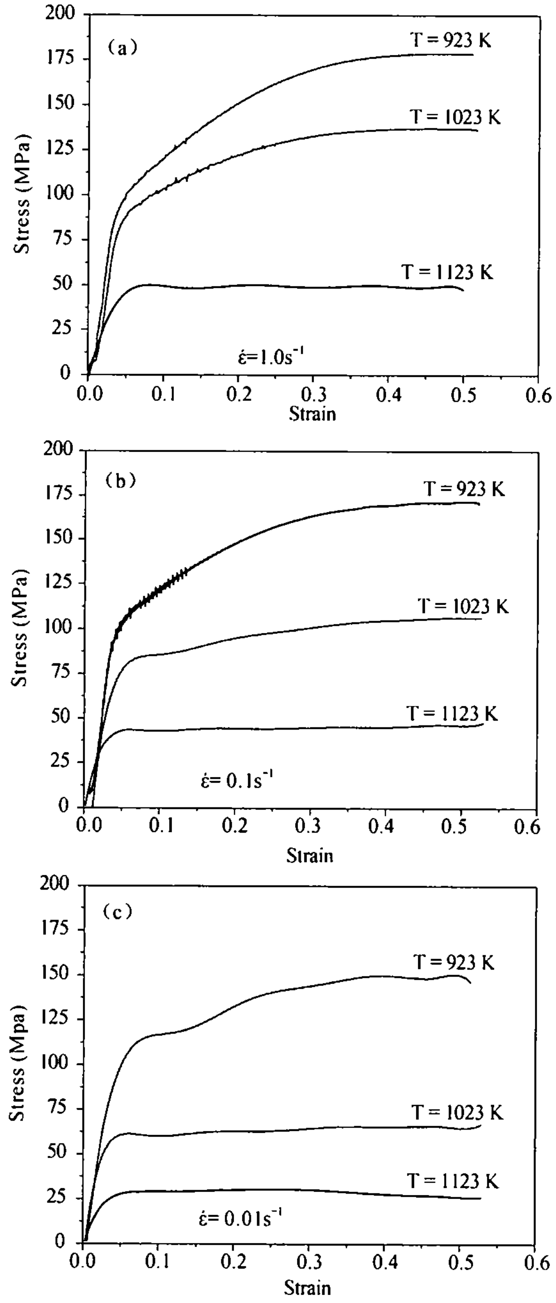

Figure 1. Typical flow curves of CP-Ti samples deformed at different conditions

In order to analyse the significance of these flow curves and to better understand the dependence of these flow curves on temperature and strain rate, a mathematical model has been used to determine the work hardening rate(θ). In this regard, the work hardening rate, $\theta = \partial\sigma/\partial\varepsilon$, is calculated from the flow curve and plotted as a function of stress as shown in Figure 2. It is evident that the work hardening curves are dependent on the conditions of deformation. The curves can be divided into two different types of three-stage and single stage work hardening, based on their shape. In the first type, the curves show three-stage work hardening behaviour. In the first stage, the work hardening rate decreases rapidly with increasing stress, possibly due to the dynamic recovery, until the initiation of the second stage. At this point an increase in the work hardening rate occurs and continued to a peak. In the final stage the work hardening rate decreases to a negative value, which implies work softening, which could be due to restoration mechanisms such as DRX and/or DRV and/or thermal softening

(adiabatic heating) of the sample. The three-stage work hardening behaviour was mostly observed after deformation at 923K for all deformation strain rates as shown in Figure 2.

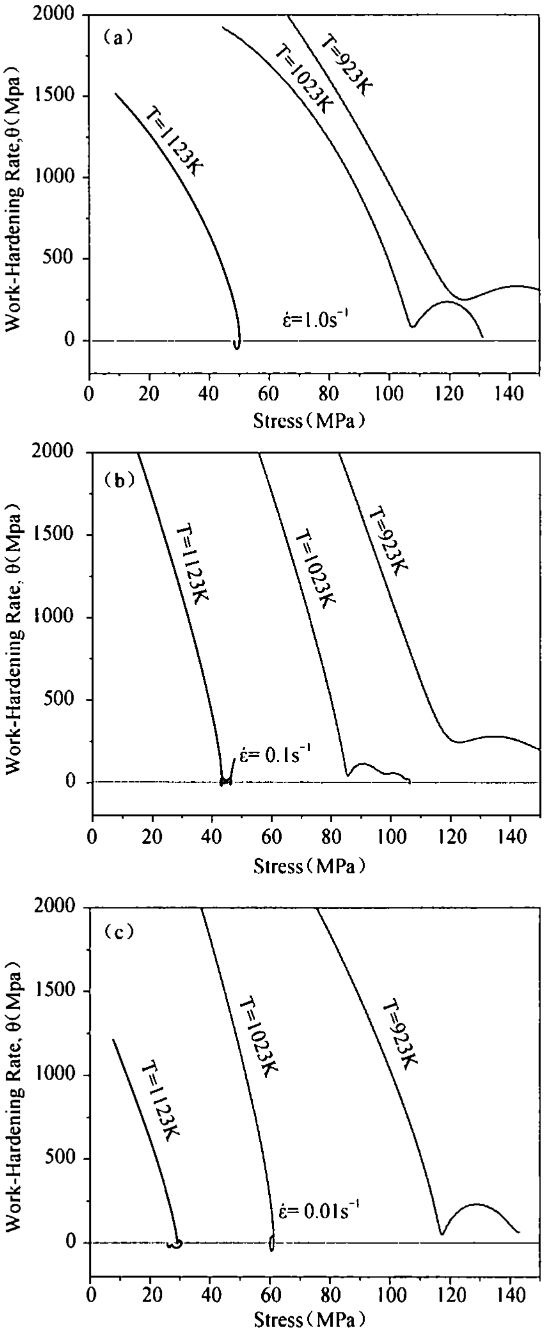

Figure 2. Work hardening rate as a function of flow stress

For single-stage work hardening, the work hardening rate continuously decreases with a very sharp slope. This indicates a high rate of work softening, which is likely due to the start of DRV and DRX. The single stage work hardening curves were observed after deformation at 1123 K(Figure 2 b and c), where the kinetics of DRX are likely to be very high due to the high mobility of Ti atoms. An earlier study[5] has shown that a two-stage work hardening behaviour can occur. In this instance, the work hardening rate rapidly decreased to a low value in the later stages of deformation.

2) Double hit deformation

In order to examine softening and microstructure development following hot-deformation, double-hit deformation tests were performed at different temperatures and strain rates. It is well documented that in steel and other metallic materials[7] upon annealing at high temperature, some degree of softening can occur. In fcc and hcp materials, this softening could be due to static recrystallization(i. e. nucleation and growth of new grains in the deformed structure)or meta-dynamic recrystallization(i. e. growth of nuclei, which are generated during deformation and had no opportunity to grow during deformation).

Figure 3 shows typical examples of double-hit deformation flow stress curves. The curves indicate that the flow stress of the deformed microstructure is significantly decreased during unloading, and the second-hit curve shows more softening by increasing the unloading time(Figure 3a). Post-deformation softening including static and meta-dynamic recrystallization has usually in the literature been held responsible for the softening behaviour of a deformed material. The occurrence of recrystallization is also expected in the current study(at least in the α-phase). To estimate the degree of softening during un-loading, its fraction is determined using the offset flow stress method and the following equation:

(1)

σ_m is the stress at the completion of the first deformation, σ_1 and σ_2 are the 0. 2 pct offset yield stress for the first and second deformations, respectively. This index of softening (X) is superimposed in Figure 3. This shows that softening fraction is increased by increasing the un-loading time(this observation is due to more time available for recrystallization to proceed) and by increasing the deformation temperature(due to increasing the kinetics of thermally-activated recrystallization processes). The result of such recrystallization processes could be grain refinement of the deformed microstructure.

3. 2 Microstructures

Figure 4 shows some examples of non-deformed, single-hit and double-hit deformed microstructures. The microstructure just before the start of deformation (after soaking at 1223 K and slow cooling to 923 K, Figure 4a)comprise very large grains of β-phase(which transformed to martensite during quench) as well as some large grains and lathes of the α phase. This microstructure indicates that almost 40% of the initial β-phase has transformed to α during cooling from the single phase β-region to 923 K. This volume fraction of transformed α was less for samples cooled to 1023K and 1123 K. This microstructure is henceforth considered to be the initial microstructure for further deformation studies. The presence of both α-and β-phases can

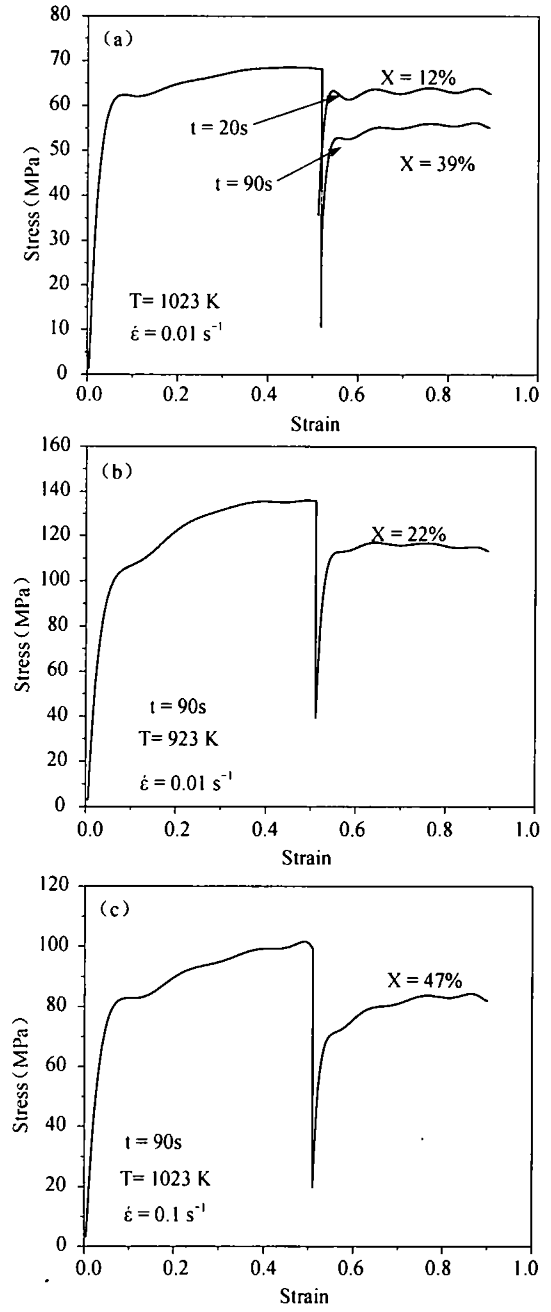

Figure 3. The flow curves of double hit deformed samples

cause different responses to deformation. The β-phase with its high stacking fault energy displays low DRX kinetics but high recovery kinetics. On the other hand, DRX can easily occur in the α-phase(hcp crystal structure). The occurrence of deformation twining is also expected in the α phase.

Figure 4b shows the microstructure of samples after deformation at 923 K. It shows that the β-phase is mostly elongated along the deformation direction and then transformed to martensite after quenching. The α-phase is recrystallized and some small and elongated α-lathes were formed. However, the formation of new α-phase due to strain induced phase transformation is also a possibility. This newly transformed α is formed as small grains or lathes along the initial β-grain boundaries.

An interesting microstructure results from the double-hit deformed samples as shown in Figure 4c. Several small and equiaxed grains of α-phase are formed during the unloading of deformed samples through meta-dynamic recrystallization. MDRX causes some softening as showed in Figure 3.

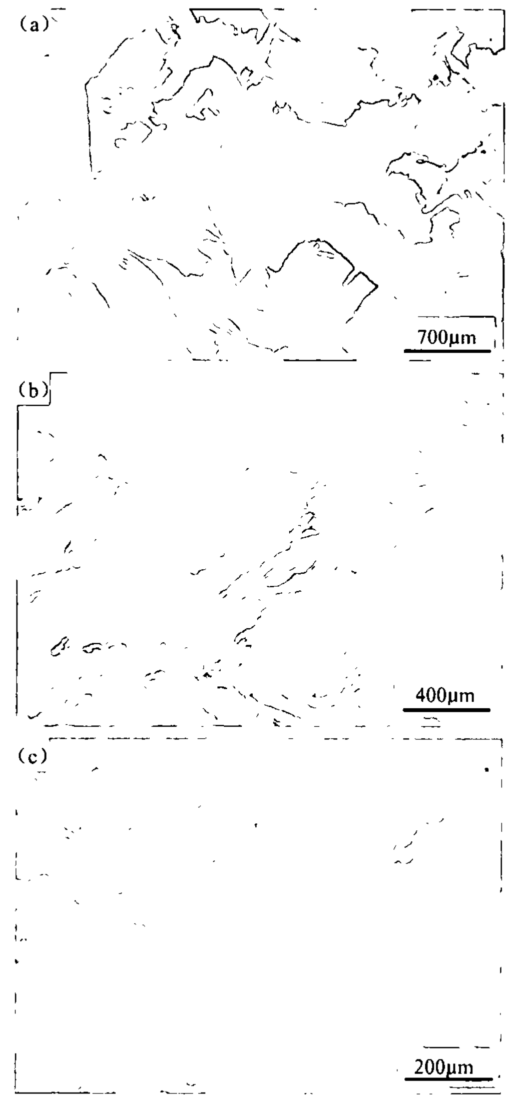

Figure 4. Microstructure of samples after quenching, (a) initial microstructure after soaking at 1223 K and slow cooling to 923K, (b) deformed at 923 K and a strain rate of 0.01 s^{-1}, (c) double hit deformed at 1023 K, strain rate of 0.01 s^{-1} and an un-loading time of 90 s

4. Conclusions

The microstructure and flow stress behaviour of commercially pure titanium alloys were studied during and following hot deformation using single and double-hit deformation tests. The flow stress is strongly dependent on the strain rate and temperature. At higher temperatures, the flow curves indicated a typical dynamic recrystallization response, while at lower temperatures continuous work hardening occurred. Double-hit deformation tests confirmed the occurrence of work softening in CP Ti following deformation at high temperature. The softening fraction increases as a function of time and strain rate. This softening is accompanied by meta-dynamic recrystallization of the deformed samples.

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REFERENCES

1) D. R. Chichili, K. T. Ramesh and K. J. Hemker, Acta Mater. 1998;46:pp. 1025-1043.

2) A. Dehghan-Manshadi, M. R. Barnett and P. D. Hodgson, Mater. Sci. Eng. A. 2008;485A:pp. 664-672.

3) A. Dehghan-Manshadi, M. R. Barnett and P. D. Hodgson, Metall. Mater. Trans. A. 2008;39A:pp. 1359-1370.

4) S. Nemat-Nasser, W. G. Guo and J. Y. Cheng, Acta Mater. 1999; 47:pp. 3705-3720.

5) Z. Zeng, Y. Zhang and S. Jonsson, Mater. Des. 2009;30:pp. 3105-3111.

6) R. E. Reed-Hill, C. V. Iswaran and M. J. Kaufman, Script. Metall. Mater. 1995;33:pp. 157-162.

7) A. Dehghan-Manshadi, M. R. Barnett and P. D. Hodgson, Metall. Mater. Trans. A. 2008;39A:pp. 1371-1381.

8) T. Furuhara, B. Poorganji, H. Abe and T. Maki, JOM. 2007;64-67.