

# The Hot Deformation Behaviour of Antibacterial Ti-10wt.%Cu Sintered Alloy

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**Abstract.** Ti-10wt.%Cu sintered alloy was prepared by a powder metallurgy method to obtain strong antibacterial property. In order to optimize the deformation processing, the hot deformation behaviour of Ti-10wt.%Cu sintered alloy was investigated in this paper. The deformation temperature was set between 800°C to 1000°C and the strain rate was set in a range of  $0.008\text{ s}^{-1}$  to  $5\text{ s}^{-1}$ . The microstructure of the deformed alloy was observed in order to reveal the effect of the hot deformation process on the microstructure. The results showed that the Ti<sub>2</sub>Cu phase distributed homogeneously with the increasing of the deformation temperature and decreased with the increasing of the strain rate. It was concluded that the final optimized deformation process was: a deformation temperature range of 850°C to 950°C, and a strain rate of  $0.1\text{ s}^{-1}$  to  $0.5\text{ s}^{-1}$ .

## 1. Introduction

Titanium and titanium alloys have been widely used as tissue repair materials for clinical treatment due to the good corrosion resistance, mechanical property and biocompatibility. It was reported that surgical site infection represents the most common nosocomial infection, accounting for 15% of all infections[1]. Pastides P. found periprosthetic infections in orthopaedics occur with a high frequency in primary hip and knee arthroplasties[2]. The main reasons are the colonization of bacteria on the materials surfaces and the formation of biofilm, showing a 500-5000 times more resistant to antimicrobials as compared to planktonic bacteria[3, 4]. In our previous studies, the Ti-Cu sintered alloy showed very strong antibacterial property against *S. aureus* and *E. coli*, and good biocompatibility and corrosion resistance[5-7]. Despite of those advantages, the brittleness of the sintered Ti-Cu alloy limits its uses. Hot extrusion is an effective method to improve the ductility of sintered titanium alloy by homogenizing the microstructure and eliminating the microstructure defects[8]. Recently, some research was reported on the deformation behavior of titanium alloy. For example, Bai et al[9] studied the deformation behaviour of a TLM alloy (Ti-3Zr-2Sn-3Mo-25Nb) and obtained a most peak efficiency domain at around 850°C. In Qin's research[10], the high-temperature flow behaviour of TC11/Ti-22Al-25Nb weldments was investigated in detail and the hot working processing could be carried out safely in the domain with a temperature range of 900-1060°C and a strain rate of  $0.001\text{--}0.6\text{ s}^{-1}$ . Thus, whether the Ti-10wt.%Cu sintered alloy shows good antibacterial property after hot deformation has high research value.

The aim of this work was to optimize the deforming parameters. The hot deformation behaviour of Ti-10wt.%Cu sintered alloy was studied by an isothermal compression test in a temperature range of 800°C~1000°C with a strain rate of  $0.004\text{--}1\text{ s}^{-1}$ . Based on the true stress and the true strain curves, a constitutive equation was established.



## 2. Methods

### 2.1. Preparation of Ti-10wt.%Cu Sintered Alloy

High pure Ti and Cu powder were mixed for 24h with a nominal composition of Ti-10wt.%Cu in a ball mill. The mixed powder was then cold pressed by 300MPa and high pressure sintered at 1000°C for 3h under 35MPa in a graphite moulds to obtain a cylindrical Ti-10wt.%Cu ingot with 30 mm in height and 40 mm in diameter. Samples for the compressive test with a dimension of 12 mm in height and 8 mm in diameter were sliced from the sintered ingot by a discharge method.

### 2.2. Isothermal Compressive Test

All samples before test were ground carefully by SiC papers up to 2000 grits and polished by 1 $\mu$ m polishing paste. The isothermal compressive strain test was conducted on a MMS-200 machine thermal simulator at temperatures of 800°C, 850°C, 900°C, 950°C and 1000°C, maximum true strain of 0.6, and strain rates of 0.004s<sup>-1</sup>, 0.02s<sup>-1</sup>, 0.1s<sup>-1</sup>, 0.5s<sup>-1</sup> and 1s<sup>-1</sup>.

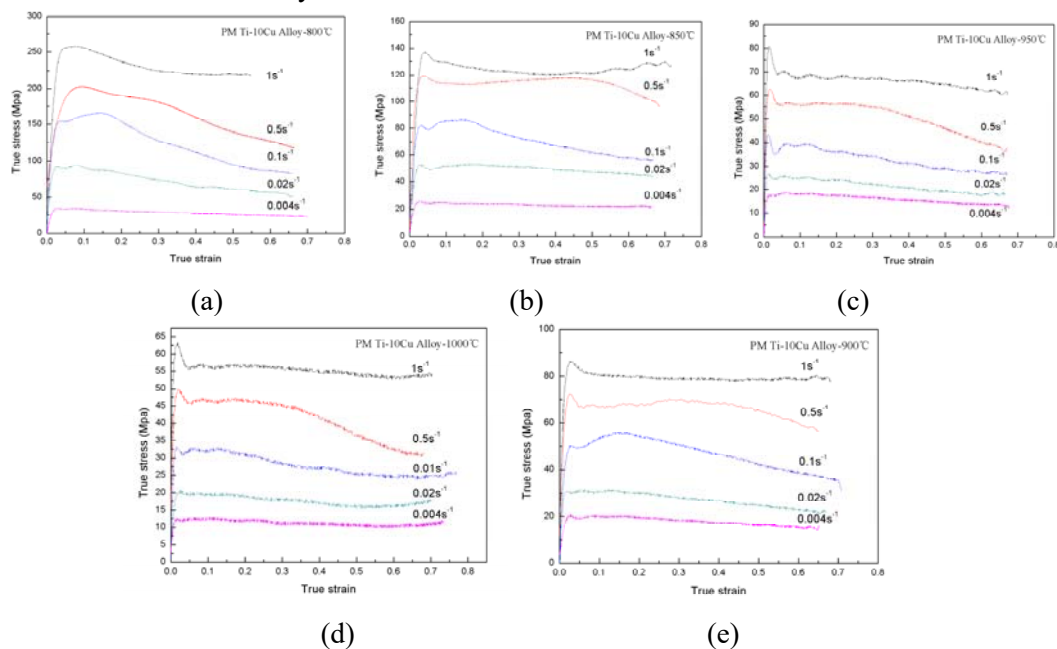
### 2.3. Microstructure

Samples before and after compression were sliced along the longitudinal direction for the microstructure examination. Metallographic samples were ground and polished as described above, and then were etched in a mixed solution of 5% HF+15% HNO<sub>3</sub>+ 80% alcohol. Microstructure was observed by optical microscope and scanning electron microscope (SEM).

## 3. Results

### 3.1. Stress Flow Behavior of Ti-10wt.%Cu Alloy

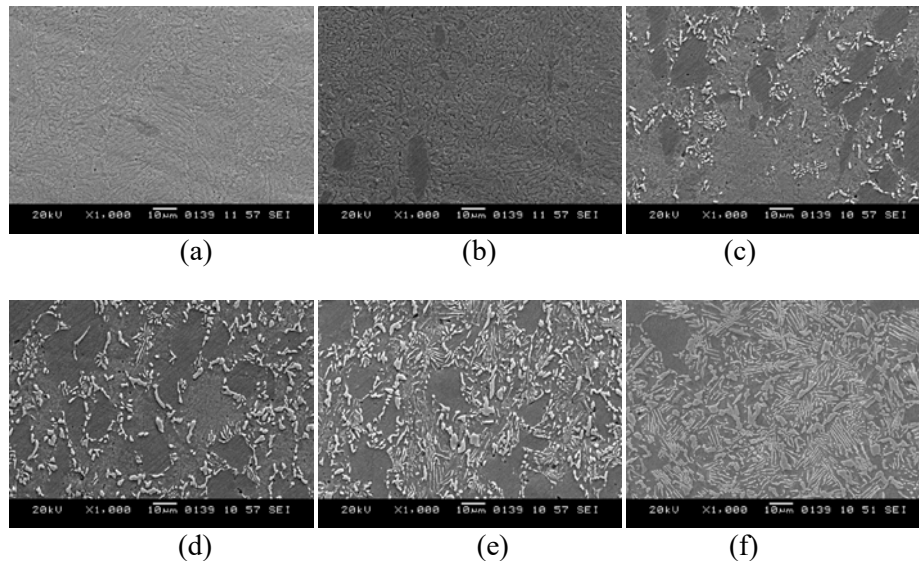
The intrinsic relationship between the flow stress and the thermal-dynamic behaviour can be reflected from the true stress-true strain curves, as shown in Figure 1. The flow curves were consisted of the following steps: elastic deformation, work hardening, softening and steady deformation. The flow stress showed an obvious decrease trend with the increasing of the deformation temperature and the decreasing of the strain rate. The stress increased to a peak at the initial small strain due to the effect of work hardening due to the fast increase of flow stress and generation of dislocation, then the flow stress decreased until a relatively stable stress.



**Fig.1** Typical true stress-true strain curves of Ti-Cu alloy obtained at temperatures of (a) 800°C; (b) 850°C; (c) 900°C; (d) 950°C; (e) 1000°C

### 3.2. Microstructure

Figure 2a shows the undeformed microstructure of sintered Ti-10wt.%Cu alloy. Figure 2b~f show the microstructure of Ti-10wt.%Cu alloys deformed at different temperatures with a strain of 0.6 and strain rate of  $0.004\text{s}^{-1}$ . Typical  $\text{Ti}_2\text{Cu}$  phase (lamellar-type) and Cu-rich phase (block-type) distributed discontinuously in the matrix (as shown in Figure 2a) which were systemically studied in our previous research[5]. After deformation, the grain was elongated along the vertical deformation direction and the size of secondary phase ( $\text{Ti}_2\text{Cu}$  phase and rich copper phase) were decreased. Even when the deformation temperature exceeded  $950^\circ\text{C}$ , the secondary phase showed fine size and distributed homogeneously in the matrix.



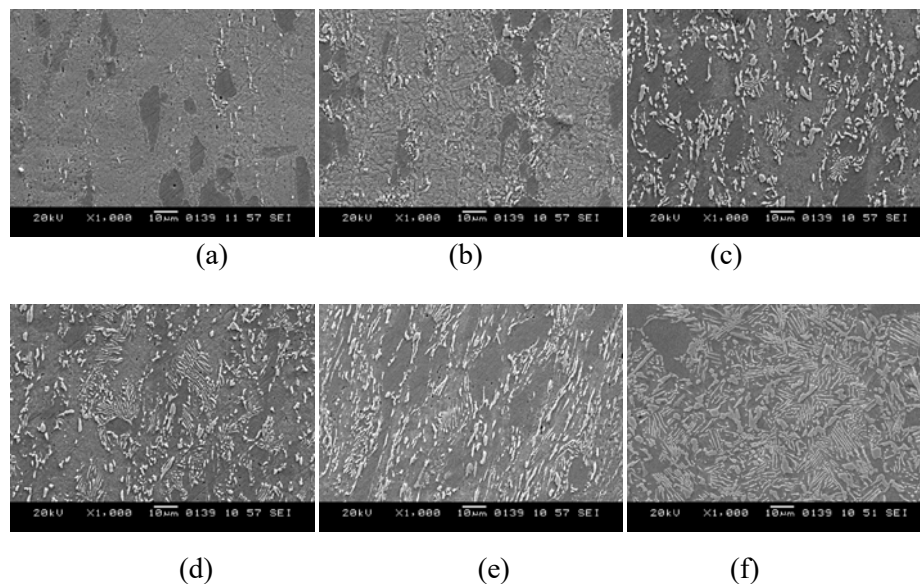
**Fig. 2** The vertical microstructure of the deformed Ti-10wt.%Cu alloy at a strain rate of  $0.004\text{s}^{-1}$  and different temperatures ( $\epsilon=0.6$ )

(a) undeformed alloy; (b)  $800^\circ\text{C}$ ; (c)  $850^\circ\text{C}$ ; (d)  $900^\circ\text{C}$ ; (e)  $950^\circ\text{C}$ ; (f)  $1000^\circ\text{C}$

At  $800^\circ\text{C}$ , original lamellar-type  $\text{Ti}_2\text{Cu}$  phase was obviously elongated and bended, obviously dynamic recrystallization was not observed. With the increasing in the deformation temperature, thermal vibration and diffusion rate of atom increase, Cu atom diffused fully to matrix Ti, which lead to the increase of  $\alpha\text{-Ti}$  phase domain size. When the compression temperature increases to  $950^\circ\text{C}$ , Cu-rich phase become spherical and segregate to  $\alpha\text{-Ti}$  phase boundaries. When temperature increase to  $1000^\circ\text{C}$ , recrystallization microstructure became homogeneous and grain size become larger, indicating complete recrystallization occurred in alloy. It is can assumed that  $\alpha\text{-Ti}$  phase domain became coarse because Cu diffuse to matrix at the temperature of  $900^\circ\text{C}$  and no secondary  $\text{Ti}_2\text{Cu}$  phase was observed at  $950^\circ\text{C}$  and  $1000^\circ\text{C}$ .

Figure 3a~e show the microstructures of the alloys deformed at different strain rates. The deformation temperature was  $900^\circ\text{C}$ . The grain was also elongated along the vertical deformation direction and the size of secondary phase ( $\text{Ti}_2\text{Cu}$  phase and rich copper phase) were decreased with the decreasing of the deformation rate. When the deformation was reduced to  $0.004\text{s}^{-1}$ , nearly all the secondary phases were crushed and presented as tiny shape (as shown in Figure 3e). At the strain rate of  $0.004\text{s}^{-1}$ ,  $\alpha\text{-Ti}$  phase exhibited approximately ellipse. The recrystallized  $\text{Ti}_2\text{Cu}$  phase can grow adequately due to the enough long deformation time, so recrystallization and growth were more obvious than with the strain rate of  $1\text{s}^{-1}$ . When strain rate increased to  $1\text{s}^{-1}$ ,  $\alpha\text{-Ti}$  phase became larger and grew along compressing direction, and Cu-rich phase increased. Original microstructure of the alloy showed obvious orientation, lamellar-type  $\text{Ti}_2\text{Cu}$  phase was distributed along the direction vertical to the compression direction. the abnormal growth of Cu-rich phase was observed and partial spherical Ti-Cu phase (maybe  $\text{Ti}_2\text{Cu}$ ) was found along Cu-rich phase boundary. The main reason

might be due to that more deformation energy was accumulated in alloy, As reported, the presence of Ti-Cu intermetallic phase would lead to a relatively weak bond due to their brittle nature, so we supposed that the mechanical property can be improved after hot compression and the specific mechanical property need to be studied in our next research.



**Fig.3** The vertical microstructure of the alloys deformed at 900°C and with a deformation of 0.6 and at different strain rate.

(a)  $1\text{ s}^{-1}$ ; (b)  $0.5\text{ s}^{-1}$ ; (c)  $0.1\text{ s}^{-1}$ ; (d)  $0.02\text{ s}^{-1}$ ; (e)  $0.004\text{ s}^{-1}$  (f) undeformed alloy

#### 4. Conclusions

The hot deformation behaviour and the hot processing maps for antibacterial Ti-10wt.%Cu sintered alloy have been studied at a temperature range of 800°C~1000°C with a strain rate of 0.004~ $1\text{ s}^{-1}$ . The following conclusions were obtained from the investigation:

- (1) The instability region occurred at high strain rates ( $>0.5\text{ s}^{-1}$ ) and low temperatures ( $<850^\circ\text{C}$ ). It was suggested that this region should be avoided during the hot deformation processing for the Ti-10wt.%Cu alloy. The optimal working region obtained from the processing map appears at 850-950°C/ $0.1\text{--}0.5\text{ s}^{-1}$  and the efficiency of power dissipation was 45-50%.
- (2) Ti-10wt.%Cu alloy obtain homogenized microstructure after hot deformation.

#### 5. Acknowledgments

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