Effect of Surface Treatment on the Hot Forming of the High Strength Ti-6Al-4V Fastener

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The Ti-6Al-4V alloy is a very attractive material for the fastener bolt of an aircraft because of its excellent combination of specific strength and corrosion resistance. However, poor workability and severe galling between work-piece and die material make it difficult to produce. The aim of the present work is to enhance the hot formability of the Ti-6Al-4V bolt. Multiple forgings were simulated using FE code to determine optimum process parameters including workpiece temperature, strain rate, and damage value. Various thermal oxidation treatments were investigated in order to prevent galling and increase the hot-workability. After the oxidation treatment, hot forging was conducted and the microstructure of forged specimens was investigated. Among the various heat treatments, holding at 750°C for 2 h provides the most homogeneous microstructure and a sufficiently low galling phenomenon after forging. Mass-gain versus time curves for isothermal oxidation were obtained in order to analyze the oxidation behavior. [doi:10.2320/matertrans.M2009113]

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1. Introduction

For application to the aerospace industry, the fastener part must be carefully designed and manufactured in a sophisticated process.¹⁾ These days, titanium fasteners have become attractive for use in aircrafts because of their high specific strength and corrosion resistance.^{2,3)} The titanium alloy will also be more attractive with the rise of oil prices. So far, most titanium alloys used for the fastener part are commercial pure titanium and β -titanium alloy⁴⁾ since they can be easily forged at room temperature. Meanwhile, the Ti-6Al-4V alloy, which has ~ 1100 MPa tensile strength under solution and an aging heat-treatment condition, is not widely applied because of its poor workability. Usually the Ti-6Al-4V bolt is manufactured by the following process: hot forging $\rightarrow$ heat treatment $\rightarrow$ machining (including lathe turning and thread rolling).⁵⁾ In particular, the hot forging process requires a very complicated procedure, unlike cold-forging. For example, the temperature of the workpiece, distribution of the accumulated strain, and processing time must be carefully controlled. Besides that, the titanium alloy easily galls to the die materials since it is originally a very active metal.⁶⁾ Thus, a passive layer should be coated to the Ti-6Al-4V surface to prevent galling. The aims of the present work are to develop the Ti-6Al-4V bolt forging process and to investigate the effect of the passive layer on galling.

2. Experimental Procedure

The material used was a hot rolled Ti-6Al-4V bar with a chemical composition of Ti-5.8Al-4.18V-0.02N-0.10O-0.05Fe (in wt pct.). The as-received material was solution treated at 927°C for 2 h, followed by furnace cooling to room temperature. The resulting microstructure was an equiaxed- α microstructure with grain sizes of $\sim 8\mu\text{m}$ (Fig. 1). Tensile strength and elongation of the material were 963 MPa and 22%, respectively.

Total accumulated strain and Cockcroft-Latham damage values⁷⁾ were calculated by a finite element simulation code.

Fig. 1 Microstructure of Ti-6Al-4V alloy after solution treatment.

The shape of punch and bottom die were designed on the basis of the FE-simulation. The punch and die material was ASTM H13 (JIS SKD61) tool steel. The processing parameters used in the simulation are given in Table 1. In this process, a thermo-viscoplastic material model was used for the workpiece and a thermo-rigid material model for the punch and die material. The flow stress versus strain relationship was experimentally obtained by a hot compression test.

In order to prevent the galling between workpiece and die, the as-received material was thermally oxidized at various temperatures. Before the thermal oxidation, workpieces were cut to bolt size and cleaned ultrasonically in acetone and ethanol prior to oxidation. After the oxidation, the workpieces were heated to 600°C and then immediately transferred to a bolt former machine which contains 4 stage forging dies. After final forging, the workpiece was cross-sectioned and analyzed with an optical microscope. Isothermal oxidation tests in dry air were performed in a Setaram (Caluire, France) at 600°C, 750°C, and 850°C. The heating rate was 50 K/min and the temperature deviation during the tests was smaller than $\pm 1^\circ\text{C}$. From

Table 1 Physical properties of materials and process conditions used in present work.

Physical properties of materials		
	Ti-6Al-4V	ASTM H13
Density (kg/m ³)	4300	7760
Coefficient of thermal expansion (°C ⁻¹)	8.8×10^{-6}	11.5×10^{-6}
Heat capacity (N/mm ² °C)	18.0	28.6
Heat transfer coefficient between workpiece and die/punch (N/°C s mm)	11	11
Emissivity	0.1	0.1
Process conditions		
Punch speed	200 mm/s	
Workpiece temperature (°C)	600	
Die/Punch temperature (°C)	200	
Lubricant	Oil base graphite

Fig. 2 Processing map constructed on the basis of the shape of compressed specimen. Symbol 'x' denotes surface cracking, '▲' unstable deformation, and '●' sound deformation, respectively.

the isothermal oxidation test, mass-gain versus time curves were obtained.

3. Results and Discussion

3.1 Finite element simulation of bolt forging process

The finite element simulation of the bolt forging process was performed using commercial FE-code, DEFORM-2D. The flow stress versus strain curves obtained from the hot compression test were incorporated in the finite element code. The hot forging condition was selected by considering the forged shape of the hot compression specimen. Figure 2 indicates the processing map of the Ti-6Al-4V alloy used in the present work, where the symbol 'x' denotes surface cracking, '▲' unstable deformation, and '●' sound deformation, respectively. From the processing map, the temperature of 600°C and strain rate of 1.0 s^{-1} were proposed as an appropriate forging condition because they provide the highest forging speed at the lowest temperature. Figure 3 shows the finite element simulation results performed at the proposed condition. The distribution of the Cockcroft-Latham fracture criteria for bulk forming was calculated as shown in Fig. 3(e). The maximum value of 0.152 was obtained at the outside bottom corner of the head part. Such a

(a) 1st forging stage(b) 2nd forging stage (c) 3rd forging stage(d) 4th forging stage (e) After 4th forging (f) After 4th forging

Fig. 3 Forging sequence and distribution of deformation characteristics of hot forged workpiece: (a) 1st stage forging, (b) 2nd stage forging, (c) 3rd stage forging, (d) 4th stage forging, (e) distribution of Cockcroft-Latham damage value after 4th forging, (f) distribution of maximum principal stress after 4th forging.

Fig. 4 Macrograph of Ti-6Al-4V bolt forged without any surface treatment. Arrows indicate the location of galling.

value is found to be moderate compared to the previous report by Semiatin *et al.*⁸⁾ Since the area with the highest damage value is designed to be machined out, the remaining finish parts are thought to be unaffected by the highly damaged area.

3.2 Effect of thermal oxidation on the workability

In order to reduce the friction stress between workpiece and die material, four different lubricants (oil-base graphite, MoO₂, Boron-Nitride, and phosphate coating) were tested. Among them, the oil-base graphite lubricant exhibited the most effective performance while others showed significant galling. However, despite that, the lubricant by itself did not prevent galling sufficiently. The occurrence of galling is mainly due to the high reactivity of titanium with the other material. This high reactivity results in extensive local adhesion, surface distortion, and material transfer during forming. Thus, fresh and clean titanium should not be in direct contact with the die material during the specific surface treatment.⁹⁾ Hutchings *et al.* investigated six different surface treatments for the galling protection of titanium alloys.¹⁰⁾ Diamond-like carbon coating and thermal oxidation were found to provide excellent protection against substrate galling. In the present work, the thermal oxidation method was proposed because of its convenience and cost effectiveness.

As a trial, a titanium bolt was forged without any surface treatment. The trial-production of the Ti-6Al-4V bolt is shown in Fig. 4, where arrows indicate the galling developed location. Due to the galling, the workpiece was seized with dies and did not eject well at each step. Microstructures of the cross-sectioned area are shown in Fig. 5. Although the internal part had a sound equiaxed microstructure after forging (Fig. 5(a)), intense shear bands were noticed at the head part due to galling (Fig. 5(b) and (c)). Moreover, surface cracking occurred at the outside corner area of the head part (Fig. 5(d)) where the maximum stress level of above 1400 MPa was loaded. Although the maximum Cockcroft-Latham damage value at this region is not so high (<0.1), some cracks are observed here. This is because high friction stress and local adhesion could induce the crack initiation in the surface region. The Cockcroft-Latham fracture criterion is based on void initiation and thus not adequate for a surface friction problem.

In order to mitigate the galling, the workpiece was exposed to 927°C, 850°C, 750°C, and 600°C for 2 h at dry air and then air cooled. Final forged shapes of each material are shown in Fig. 6. Although galling did not occur at all, the workpiece with the thermal oxidation at 927°C had very poor workability because the initial equiaxed microstructures transformed to a Bi-modal microstructure, which has a relatively lower ductility and higher strength than the equiaxed one.¹⁾ Because of the poor workability, the hot forging process was not completed and was stopped at the 3rd stage of forging. Besides that, the brittle α -case with the thickness of above 50 μm enhanced crack initiation at the surface region as shown in Fig. 7. The α -case has a tetragonal rutile crystal structure of which deformation mode is a planar not a wavy slip.¹¹⁾ Therefore, the hard, less ductile α -case can result in the formation of surface cracks under tension loading. Thus, the thickness of α -case must be carefully controlled not to exceed the appropriate thickness which will be discussed in Section 3.3.

On the other hand, the workpiece with an 850°C heat treatment (Fig. 6(b)) exhibited mild formability and good surface quality. Microstructural homogeneity was better than that of the non-oxidized workpiece as shown in Fig. 8. Intense shear bands and dead-metal zone are hardly observed. However, still some surface cracks are noticed on the α -case (Fig. 8(d)). Thickness of the retained α -case was about 23 μm which could induce crack initiation.

As for the workpiece with a thermal oxidation at 750°C, it revealed a shiny surface and excellent formability (Fig. 6(c)). Two hours of exposure at 750°C provided a thin dense TiO₂ layer which could efficiently prevent galling. Figure 9 shows that its microstructure remained as a fine equiaxed one (Fig. 9(a)) and no surface cracks or intense shear bands were found (Fig. 9(b) and (c)). And, most of the α -case was worn out after the final forging due to friction with the die material. The thickness of the retained α -case was measured to be below $\sim 5 \mu\text{m}$ (Fig. 9(d)).

Similar to the uncoated workpiece, the workpiece thermally oxidized at 600°C was galled and not ejected well from the 3rd step dies (Fig. 6(d)). Therefore, the workpiece was removed from the dies manually and then heated again to continue final forging. It is thought that if the initial thickness of the oxide layer is too thin, then it will be easily worn out at an early stage of forging, and galling might occur at the next stage. Hence, for galling protection, a minimum thickness of oxide layer must be coated.

For the best formability, both the galling protection and low friction coefficients should be provided at the same time. Normally surface oxide films on most metal surface deteriorate the lubricity in forming. However, in this case, the thermal oxidation treatment seems to reduce the friction between Ti-6Al-4V and the dies material. In previous research,¹⁰⁾ it was found that the thermally oxidized titanium alloy exhibited a lower friction coefficient than uncoated titanium after initial loading stage. The primary cause for this finding is thought to be that the friction coefficient is affected by not only the surface roughness but also local adhesion and material transfer in the contact region.

Fig. 5 Microstructural observation of Ti-6Al-4V bolt forged without surface treatment.

Fig. 6 Macrographs of forged Ti-6Al-4V bolt which was thermally oxidized at (a) 927°C, (b) 850°C, (c) 750°C, and (d) 600°C before forging.

Fig. 7 Micrograph of forged Ti-6Al-4V bolt which was thermally oxidized at 927°C before forging. Surface cracks were initiated at thick α -case region (gray colored area).

3.3 Thermal oxidation rate at 600°C, 750°C, and 850°C

Mass-gain versus time curves for isothermal oxidation in the air at temperatures from 600°C to 850°C are shown in Fig. 10. At all temperatures, the oxidation kinetics didn't follow the parabolic rate law which is usually seen in other titanium alloy systems¹²⁾ up to 10 h. The temperature dependency of the oxidation rate constant was quite considerable. In particular, the amount of mass-gain at 850°C was much higher than that at lower temperatures. After 2 h of exposure at 850°C, the total mass-gain amount is about 11 mg/cm², which is enough to make the surface brittle. Thus, thermal exposure needs to be performed below 850°C. As for the result at 600°C, the amount of mass-gain was below 0.2 mg/cm² up to 10 h, which is too small to prevent galling. Considering the results in Section 3.2, exposure at 750°C, which provides a TiO₂ oxide layer of 0.8 mg/cm², seems to be suitable for the thermal oxidation treatment. However, this amount of oxidation layer does not guarantee the galling protection for all other kinds of forging because the galling phenomenon depends on many factors such as load pressure, dies material, and forging condition.

Fig. 8 Micrographs of forged Ti-6Al-4V bolt which was thermally oxidized at 850°C.

Fig. 9 Micrographs of forged Ti-6Al-4V bolt which was thermally oxidized at 750°C.

Fig. 10 Mass-gain versus time curves for isothermal oxidation of Ti-6Al-4V alloy in dry air at 600°C, 750°C, and 850°C.

3.4 Tensile properties after solid solution and aging treatment

After forging, a solid solution and aging treatment was applied to improve the room temperature mechanical properties. In order to balance the strength and ductility, various heat-treatment effects were investigated. Holding time and cooling methods of solid solution and aging treatment were summarized in Table 2. It is noticed that the tensile strength increases and elongation decreases with the increase of water quenching temperature because the microstructure morphology change from equiaxed to Bi-modal. For the aging treatment, it gives a much higher yield strength and lower ductility to the Ti-6Al-4V alloy due to the precipitation of coherent α_2 particles.^{11,13)} Comparing heat-treatment 'A' and 'B', the effect of the aging treatment on tensile strength and elongation was not significant. However, the yield strength was noticeably distinguished according to the aging treatment. Even though the largest elongation was exhibited in heat-treatment 'C', this heat-treatment was discarded because the resulting tensile and

Table 2 Room temperature tensile properties as a function of solid solution and aging treatment.

Heat-treatment		Tensile properties		
Solid solution	Aging	Yield strength (MPa)	Tensile strength (MPa)	Elongation (%)
A	927°C for 1 h/WQ	880	1142	18.7
B	927°C for 1 h/WQ	1067	1183	17.2
C	927°C for 1 h/FC to 720°C/WQ	726	963	22.3
D	927°C for 1 h/FC to 720°C/WQ	938	1011	20.6
E	927°C for 1 h/FC to 900°C/WQ	1074	1136	19.2
F	927°C for 1 h/FC to 850°C/WQ	1059	1096	20.2

*Note: AC = air cooled, WQ = water quenched, FC = furnace cooled

yield strengths were very low. In the present work, the heat-treatment 'F' is proposed as the optimized one due to the excellent balance between strength and ductility. It provided nearly a 1100 MPa tensile strength and over 20% tensile elongation, which are usual requirements for industrial purpose.¹⁴⁾

4. Conclusions

The hot forging process of the Ti-6Al-4V alloy was established. For this purpose, a processing map was constructed to propose appropriate forging conditions. In order to verify the conditions, a finite element simulation was performed and damage factors were calculated. The effect of thermal oxidation was investigated to prevent galling between the rare Ti-6Al-4V alloy and die. Thermal exposure at 750°C for 2 h exhibited the most excellent surface quality and workability.

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REFERENCES

- 1) M. J. Donachie Jr.: *Titanium—A Technical Guide 2nd edition*, ASM International, Materials Park, (2000) pp. 5–11.
- 2) G. W. Kuhlman: *Microstructure/property relationships in titanium aluminides and alloys*, Warrendale, PA: (The Minerals Metals & Materials Society, TMS; 1991) pp. 465–491.
- 3) G. W. Kuhlman and F. R. Billman: *Met. Prog.* **111** (1977) 38.
- 4) N. Matsukura, F. Kamiyama and T. Morikawa: *J. Jpn. Inst. Light Metals* **55** (2005) 599–603.
- 5) Y. Cui, Q. Meng, B. Zhang and F. Yan: *Eng. Failure Analysis* **13** (2006) 669–672.
- 6) N. Matsukura: *Ti-2007 science and technology*, ed. by M. Niinomi, S. Akitama, M. Hagiwara, M. Ikeda and K. Maruyama, (Kyoto, 2007) pp. 1395–198.
- 7) M. G. Cockcroft and D. J. Latham: *J. Inst. Met.* **96** (1968) 33.
- 8) S. L. Semiatin, R. L. Goetz, E. B. Shell, V. Seetharaman and A. K. Ghosh: *Metall. Mater. Trans. A* **30** (1999) 1411–1424.
- 9) B. Podgornik, J. Vižintin and S. Hogmark: *Surf. Eng.* **22** (2006) 235–238.
- 10) U. Wiklund and I. M. Hutchings: *Wear* **251** (2001) 1034–1041.
- 11) G. Lütjering and J. C. Williams: *Titanium 2nd edition*, (Springer, 2007).
- 12) C. Leyens: *J. Mater. Eng. Performance* **10** (2001) 225–230.
- 13) T. Yamamoto, T. Sakuma, K. Uchida, Y. Sutou and K. Yamauchi: *Mater. Trans.* **48** (2007) 439–444.
- 14) V. N. Moiseev: *Metal Sci. Heat Treat.* **43** (2001) 73–77.