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# Microstructural characteristics and unique properties obtained by solution treating or aging in $\beta$ -rich $\alpha + \beta$ titanium alloy

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## Abstract

A  $\beta$ -rich  $\alpha + \beta$  titanium alloy, SP-700 with Ti-4.5%Al-3%V-2%Mo-2%Fe exhibits unique properties in as-solution treated condition or upon subsequent aging. Stress-induced transformation and a high value of internal friction were caused by solution treating, and the age-hardening response in SP-700 was largely accelerated compared with other heat treatable titanium alloys. The relationship between these properties and nano-scale microstructure obtained by this heat treatment is discussed. © 1999 Elsevier Science S.A. All rights reserved.

Keywords:  $\alpha + \beta$  Titanium alloy; Stress-induced transformation; Age-hardening response

## 1. Introduction

The  $\beta$ -rich  $\alpha + \beta$  titanium alloy, SP-700 with Ti-4.5%Al-3%V-2%Mo-2%Fe has superior properties to Ti-6%Al-4%V alloy such as an excellent SPF property, high fatigue strength, and much improved hot or cold workability[1,2]. These advantageous properties in SP-700 arise primarily from its microstructural characteristics and alloy composition. This alloy exhibits a wide variety of microstructures depending on heat treatment conditions, enabling itself to yield several unique properties. In particular, this alloy in as-solution treated condition gives rise to stress-induced transformation accompanied with a large reduction of yield strength, and also a high value of internal friction. Both properties were reported to be observed in several titanium alloys of  $\alpha + \beta$  [3,4] and  $\beta$  types [5,6], and they were explained based on the nature of the microstructural constituents formed by a particular heat treatment condition. The past studies indicated that stress-induced transformation was caused by transformation of unstable  $\beta$  phase to  $\alpha''$  martensite [3–5], and that high damping might be attributed to the lattice defects or twinning formed in the  $\alpha''$  martensite [6–8]. Upon aging after solution treating, this alloy exhibits a much accelerated age-hardening response. That is, the holding time needed to obtain the peak hardness at the temperature around 500°C in this alloy, is about one hour, being a much shorter aging time compared with other heat-treatable titanium alloys. This accelerated aging response may be attributed to both a microstructural feature and the composition of this alloy.

The present study deals with these properties obtained by solution treating or subsequent aging in the alloy of SP-700. The microstructures obtained by solution treating or aging were investigated in detail, including TEM observation of the nano-scale microstructural constituents such as  $\alpha'$  or  $\alpha''$  martensite, acicular  $\alpha$ , thermal  $\omega$  phase and retained  $\beta$  phase. The basic causes for these properties are discussed based on the results of microstructural analysis.

## 2. Experimental procedures

The chemical compositions of the titanium alloys used in this study are listed in Table 1. No.1 alloy of SP-700 was used for stress-induced transformation, being a 3 mm thick sheet from mill production. No.2 and No.4 alloys were used for internal friction study, and these ingots were melted in a laboratory VAR furnace, and hot rolled to 6 mm thick plate. No.3 alloy was used

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Table 1						
Chemical	compositions	of titanium	alloys	used	(wt%)	

No.	Alloy	Al	V	Мо	Fe	С	N	Н	0	Remarks
1		4.71	3.10	2.09	2.04	0.008	0.006	0.0029	0.08	Production heat
2	SP-700	4.68	3.00	1.93	2.01	0.003	0.002	0.0007	0.09	Labo. heat
3		4.56	3.10	1.88	2.02	0.01	0.01	0.0057	0.09	Production heat
4	Ti-6Al-4V	6.14	4.08	_	0.18	0.01	0.01	0.0004	0.16	Labo. heat

for aging studies, being a 20 mm thick plate from mill production. All plates and sheets were finally hot rolled in  $\alpha + \beta$  processing.

Solution treating was performed by reheating in the temperature region from 720 to 920°C in SP-700, or to 1050°C in Ti-6Al-4V for 1 h, followed by water quenching. For internal friction study, the cooling rate after reheating at 850°C was widely varied from 5 to 130°C s<sup>-1</sup> by means of air cooling, forced air cooling, oil or water quenching. For an age hardening study, aging after solution treating was performed in the temperature range from 360 to 600°C for various periods of heating time up to 48 h. Tensile testing specimens with gage length of 25 mm and width of 6 mm were machined from solution treated sheets. The specimens strained from 2 to 18% in tension testing at ambient temperature were also prepared for investigation of microstructural variations due to stress-induced transformation. Internal friction was measured by using the flexuous vibration type apparatus with frequency of around 400 Hz. The specimens with length of 100 mm, width of 10 mm and thickness of 1 mm were machined from solution treated plates. Hardness of aged specimens was measured with load of 10 kgf in Vickers hardness testing. The volume fractions of  $\alpha$  and  $\beta$ phases formed at a respective solution treating temperature were quantitatively measured in water quenched samples by both optical and SEM observations, and average chemical compositions of  $\beta$  phase were measured by SEM equipped with EDX. The microstructural constituents transformed from  $\beta$  phase such as  $\alpha''$ or  $\alpha'$  martensite,  $\omega$  phase as well as retained  $\beta$  phase were observed by thin foil electron microscope accompanied with electron diffraction analysis, and quantita-



Fig. 1. The effect of solution treating temperature on stress-strain curves.

tive measurement of these constituents was performed by X-ray diffraction analysis.

#### **3.** Experimental results

Fig. 1 shows the change of the stress-strain (S-S)curves with solution treating temperatures in alloy sheet of SP-700. The normal shape of the S-S curve is observed for solution treating at 800°C. The typical S-S curves for occurrence of stress-induced transformation are demonstrated at solution treating temperature from 825 to 875°C. These S-S curves are characterized by very low 0.2% proof strength (0.2%PS) accompanied with subsequent plastic flow curves. While the lowest value of 0.2% PS around 250 MPa is observed at a solution treating temperature of 850°C, the largest deformation accompanied with stress-induced transformation takes place at 875°C. Fig. 2 shows the effect of solution treating temperature on tensile properties, including the results of air cooled specimens from various temperatures. For air cooling, both 0.2% PS and tensile strength increase slightly with an increase of solution-treating temperature. On the other hand, for water quenching, the higher solution-treating temperature resulted in a large reduction of 0.2% PS and a slight increase of tensile strength. It is also evident from Figs. 1 and 2 that the elongation value increases slightly in such a solution-treating condition with the occurrence of stress-induced transformation.



Fig. 2. The effect of solution treating temperature on tensile properties.



Fig. 3. The effect of straining on variations of hardness of  $\alpha$  and  $\beta$  phases and the volume fraction of  $\alpha''$  and  $\beta$  phases.

Microhardness measurements of  $\alpha$  and  $\beta$  phases were performed using the specimens strained by tensile testing at ambient temperature. The specimens used were solution treated at 875°C. As shown in the bottom of Fig. 3, the hardness of  $\alpha$  phase continually increases with strain up to 18%. Hardness of  $\beta$  phase shows very small variation with straining up to 7% strain, above this 7% strain the rapid increase of hardness with the increase of strain is observed. The variation in volume fraction of  $\alpha''$  martensite and  $\beta$  phase with straining is shown in top figure of Fig. 3. The increase of strain up to 7% largely increased the volume fraction of  $\alpha''$ martensite accompanied with the reduction of the volume fraction of  $\beta$  phase.

Fig. 4 shows the effect of the solution treating or annealing temperature on internal friction in SP-700 and Ti-6Al-4V alloys. Both alloys resulted in very low value of internal friction in any annealing condition. In solution treating with water quenching, both alloys gave rise to the high value of internal friction in the temperature range from 820 to 920°C. In particular, SP-700 showed the largest value of internal friction at



Fig. 4. The effect of solution treating or annealing temperature on internal friction (IF).



Fig. 5. The effect of cooling rate on internal friction.

850°C. It has been reported that a high internal friction value obtained in titanium alloys was attributed to formation of  $\alpha''$  martensite [6–8]. It is important to note that a relatively high value of internal friction was obtained by solution treating above the  $\beta$ -transus temperature (900°C) of this alloy, its value being comparable to the peak value of internal friction obtained in Ti–6A1–4V alloy. The effect of cooling rate after solution treating at 850°C on internal friction is shown in Fig. 5. The decrease of cooling rate rapidly reduced internal friction. The microstructural changes with the decrease of cooling rate were the grain growth of primary  $\alpha$ , formation of acicular  $\alpha$ , decrease of  $\alpha''$ martensite and retained  $\beta$  phase. The last factor appears to cause the decrease of internal friction.

It was also found that a marked decrease of internal friction was caused by aging treatment even at low temperature around 100-200°C in both alloys. The aging treatment in heat treatable titanium alloys is mostly practiced in the temperature range from 480 to 550°C for strength-ductility balance. Hardness variations with aging time investigated in this temperature range were compared among SP-700, Ti-6Al-4V and Ti-15V-3Al-3Cr-3Sn alloys as shown in Fig. 6. Although aging temperatures were not always same in each alloy, it is evident that SP-700 shows a more accelerated age-hardening response with very high peak hardness in SP-700 was attained by an aging time of



Fig. 6. Hardness variations with aging time.  $\blacksquare$ , SP-700 (aging temp);  $\Box$ , Ti-6Al-4V (aging temp);  $\triangle$ , Ti-15V-3Al-3Cr-3Sn (aging temp).



Fig. 7. Hardness variations with aging temperature and aging time.

around 30 min to 1h at the temperature from 500 to 540°C. Fig. 7 shows the age-hardening behavior of SP-700 in a wide range of the aging temperatures. Aging kinetics were different over the temperature range. Age hardening was markedly accelerated for aging temperatures above 480°C, while it was much delayed below 420°C. In high temperature aging, the increase of the temperature shortened the aging time for peak age and reduced hardness at peak age.

## 4. Discussion

Properties obtained by solution treating in SP-700 are mostly associated with the nature of  $\beta$  phase or microstructural constituents formed by transformation of  $\beta$  phase. Table 2 shows the volume fraction and the chemical composition of  $\beta$  phase formed at various solution-treating temperatures. It is confirmed that the increase of the volume fraction of  $\beta$  phase accompanied with the increase of the temperature from 800 to 875°C, continuously reduced the content of β-stabilizing alloying elements such as V, Mo or Fe in  $\beta$  phase. As anticipated from variation of Mo equivalent value, the increase of solution treating temperature tends to reduce stability of  $\beta$  phase, promoting occurrence of stress-induced transformation in  $\beta$  phase as shown in Figs. 1 and 2. The  $\beta$  phase formed by reheating above 800°C is not fully retained at ambient temperature by water quenching, but it partially transformed into  $\alpha''$ martensite. The volume fraction of  $\alpha''$  increased with the increase of solution treating temperature as shown in Table 2. One of the authors reported that the acicular shaped  $\alpha''$  martensite, with a width of about 50 nm, was formed in layers with  $\beta$  phase [1].

As similar phenomenon to stress-induced transformation, it is well known that deformation or strain-induced transformation inducing superplasticity takes place in steels containing retained austenite or steels with metastable austenite [9,10]. The main difference between steels and titanium alloys is in the stress level for occurrence of these phenomena. While this stress in steels is mostly over the yield strength [9,10], it is less than half of yield strength in titanium alloys. The cause of a very low onset stress for stress-induced transformation seems to be that  $\beta$  phase may easily transform to  $\alpha''$  phase by utilizing the pre-existent neighboring  $\alpha''$ phase as a nucleus for transformation and that transformation can propagate into the  $\beta$  phase without or with very low increase of applied stress. When all  $\beta$ phase completes transformation, stress increases rapidly with straining. That is, stress-induced transformation in titanium alloys may be assisted by pre-existent  $\alpha''$ phase.

High internal friction observed in several  $\alpha + \beta$  type titanium alloys has been reported to be attributed to the lattice defects involved in  $\alpha''$  phase [6–8]. However, its detail mechanism seems not to be made clear yet. In the present result shown in Fig. 4, the peak value of internal friction was observed in solution treating at 850°C. However, the volume fraction of  $\alpha''$  phase continuously increased with the higher solution treating temperature as shown in Table 2, and thus, it may be difficult to explain the result of internal friction only by the presence of  $\alpha''$  phase. It may be possible that irreversible movement at the interface between  $\beta$  and  $\alpha''$ phases causes internal friction. In this case, internal friction value may be influenced by both volume fractions of  $\alpha''$  and  $\beta$  phases, and the peak value of internal friction at 850°C have resulted from the most optimum content of both phases.

Age-hardening after solution treating in SP-700 was confirmed by TEM observation that the extremely fine  $\alpha$  phase which precipitated in  $\beta$  phase caused age-hardening at high temperature above 480"C, while precipitates of thermal  $\omega$  phase were responsible for age-hardening at low temperature below 420"C. There may be two causes for the accelerated age-hardening

Table 2

The change of volume fraction and chemical compositions of  $\beta$  phase with solution treating temperature

Solution treating temperature (°C)	Volume fraction of $\beta$ phase at S.T. Temp. (%)	Chemical composition of $\beta$ phase	Mo eq.
800	50 (0%)*	Ti-3.8Al-6.1V-3.6Mo-3.9Fe	18.8
825	60 (14%)*	Ti-4.0Al-5.6V-3.4Mo-3.2Fe	16.3
850	70 (32%)*	Ti-3.9Al-4.9V-3.1Mo-2.7Fe	14.1
875	80 (58%)*	Ti-3.7A1-3.6V-2.7Mo-2.4Fe	12.0

\* The amount of  $\alpha''$  martensite formed by water quenching.

response obtained by high-temperature aging. One is that athermal  $\omega$  phase formed by solution treating may become a nucleus for  $\alpha$  precipitates, and the other is high diffusivity of iron in  $\beta$  phase. The former contributes to eliminate the incubation time for nucleation of  $\alpha$  phase, and then accelerates the aging progress. The latter promotes  $\alpha$  precipitation from  $\beta$  phase as well as decomposition of  $\alpha''$  martensite. The prolonged plateau of peak hardness in aging time observed in aging at 480°C may be due to overlapping of above two phenomena.

# 5. Conclusion

Properties and microstructures obtained by solution treating or subsequent aging in SP-700 were investigated, and the following conclusions were obtained.

1. Stress-induced transformation with very low onset stress was observed at the solution treating temperature from 825 to 875°C, and it was explained based on not only formation of unstable  $\beta$  phase, but also the important role of pre-existent  $\alpha''$  phase neighboring to  $\beta$ phase.

2. A high value of internal friction was observed after a solution-treating temperature of 850°C. Occurrence of internal friction was considered to be due to irreversible movement at the interface between  $\alpha''$  and  $\beta$ phases, in addition to internal friction of  $\alpha''$  phase itself.

3. Both the existence of  $\alpha''$  and  $\beta$  phases was needed for the occurrence of stress-induced transformation and a high value of internal friction observed in titanium alloys.

4. An accelerated age-hardening response with very high peak hardness was observed for aging at around 500°C. It was considered that the precipitation kinetics of  $\alpha$  phase and decomposition of  $\alpha''$  phase were accelerated by pre-existent athermal  $\omega$  phase and also high diffusivity of iron in  $\beta$  phase.

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