

INVESTIGATION ON THE INTERFACE PHASE BY TEM IN A NEWLY DEVELOPED NEAR α TITANIUM ALLOY^①

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ABSTRACT The interface phase (IFP) in a newly developed near α titanium alloy Ti-5123 was investigated by TEM. The interface phase was not found after quenching and subsequent aging at below 800 °C. After aging at 800 °C, the interface phase appeared at α/β boundaries. The M layer of the interface phase was showed to be an *fcc* and its orientation relationship may be expressed as $(001)_m // (110)_\beta$ and $[110]_m // [\bar{1}\bar{1}\bar{1}]_\beta$. The S layer of the interface phase was showed to possess hexagonal and twin related on $\{10\bar{1}1\}\langle\bar{1}012\rangle$ to the α . The element concentration gradient produced by diffusion at the boundaries under proper heating and cooling led to change of electron concentration, which probably was the main factor to create the interface phase in the titanium alloys.

Key words titanium alloy interface phase electron concentration

1 INTRODUCTION

Rhody C G and Williams J C, in 1975, had firstly observed an interface phase (IFP) between α (*HCP*) and β (*BCC*) in Ti-6Al-4V alloy by transmission electron microscopy (TEM)^[1]. Some researchers held that the interface phase affects the mechanical and fracture properties of titanium alloys^[2, 3]. So, in the past two decades, a great number of studies of it had been made, such as its microstructure, orientation, forming mechanism and so on^[4-7]. Up to now, two models of forming interface phase in titanium alloys were established. One is the mechanism of nucleation and growth by Rhodes and Paton^[8], the other is of the twin relationship between the interface phase and α phase by Margolin *et al*^[9].

This paper will give a TEM study of interface phases forming conditions, morpholo-

gy, microstructure, orientation, composition gradient and forming mechanism for Ti-5123 near α type alloy, which was newly developed by Shanghai Iron & Steel Research Institute.

2 EXPERIMENTAL

All the samples used in this study were cut from the hot-rolled plate of Ti-5123 alloy. The samples were treated at 950 °C for 1 h and quenched into water. After quenching, they were aged at different temperatures from 200 °C to 800 °C for 4 h, followed by air cooling.

The samples for TEM were thinned and polished to below 0.08 mm in chemical solutions $\text{HF}:\text{H}_2\text{O}_2:\text{H}_2\text{O}=2:3:3$ and $\text{HF}:\text{HNO}_3:\text{H}_2\text{O}=1:5:4$, then electropolished and perforated with a Tenupol apparatus in electrolyte $\text{HClO}_4:\text{CH}_3\text{OH}:\text{C}_2\text{H}_5\text{CH}_2\text{CH}_2\text{OH}=1:10:6$ at 25 V and -30 °C. The investigations were

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carried out on Hitachi H-700 and JEOL JEM-200 CX transmission electron microscope operated at 200 kV. The energy analyzing apparatus used was EDAX-9100 with JEM-200CX.

3 RESULTS

3.1 TEM Observation

The interface phase existed between α and β boundaries in the hot-rolled plate of Ti-5123 alloy is shown in Fig. 1.



Fig. 1 Ti-5123 alloy structures of the hot-rolled specimen

The interface phase was not found in the specimen by quenching. This means that the forming of the interface phase is related to cooling rate and the interface phase is suppressed by quenching. It is observed that β phase, produced from martensite decomposition, has grown and coarsened between α plates in the specimen by aging at 700 °C, but there was not any interface phase there (Fig. 2). In the specimen by aging at 700 °C and air-cooling, also no interface phase appeared, showing that the formation of the interface phase depends on heating temperature as well.

No interface phase existed after quenching and subsequently aging at below 800 °C. When aging temperature reached 800 °C, the interface phase appeared at α/β boundaries, with a thickness of 100~300 nm, as shown in Fig. 3. A careful TEM analysis indicated that

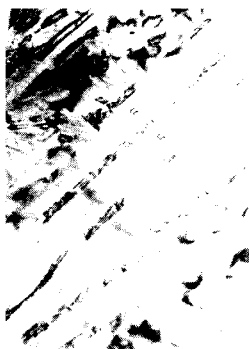


Fig. 2 Aging at 700 °C/4 h

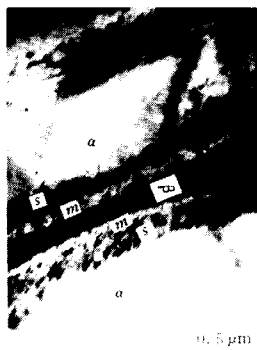


Fig. 3 Aging at 800 °C/4 h

the interface phase consisted of two layers. The monolithic layer (M layer) adjacent to α phase with smooth boundaries was identified as *fcc* structure with a relationship $(001)_m // (110)_\beta$ and $[110]_m // [1\bar{1}\bar{1}]_\beta$. Fig. 4 gives SAD pattern of M layer and the corresponding indexes. The layer adjacent to β was striated (S layer) with sawtooth boundaries and made of fine twins. The identification showed that S

layer was HCP with $\langle 10\bar{1}1 \rangle \langle \bar{1}012 \rangle$ twin relationship to α phase in Fig. 5. This result is in agree with Margolin *et al* in Ti-6Al-2Sn-4Zr-6Mo alloy^[9]. It was also found in some areas that the interface phase contained only a piece of S layer. And its dark field clearly showed the twins inserted from β into α (Fig. 6).

It should be pointed out that the samples in this study were prepared by acid solution including hydrogen in process of chemical thinning and electropolishing, and no interface phase was discovered in such samples by quenching and quenching followed by aging at below 800 °C. It means that the suggestion that interface phase is due to the preparing of TEM specimens and the interface phase is titanium hydride seems to be incorrect.

3. 2 Energy-Dispersive X-ray (EDX) Analysis

The distribution of alloying elements in α , IFP and β was measured by EDX analysis bit by bit along the direction vertical to the interface under the transmission electron microscope's STEM mode. The measured data were semi-quantitatively treated, as shown in Fig. 7. Mo content had a sharp change from α to β , and a gradient of concentration of alloying elements occurred in the area of the interface phase. This agrees with Mahajan's result in Ti-6Al-2Sn-4Zr-2Mo alloy^[10].

The same EDX analysis (Fig. 8) was completed for the specimens by aging at 700 °C, showing that the concentration of alloying elements was discontinuous at α / β .



Fig. 4 Electron diffraction pattern (a) and index (b) of the M layer at the interface phase

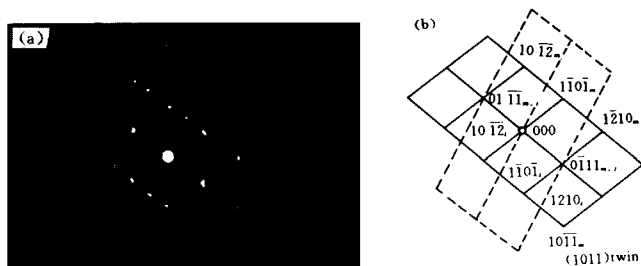


Fig. 5 Electron diffraction pattern (a) and index (b) of the S layer at the interface phase

boundaries in which the interface phase did not exist.

4 DISCUSSION

The gradient of concentration of alloying elements at α/β boundaries is controlled by the mechanism of diffusion. As we know, the diffusion depends very much on temperature, time and cooling rate. Too low temperature does not promote diffusion. And when temperature is high enough to start diffusion, a slower cooling rate is preferred to forming the gradient. If the treatment process consists of a combination of a high temperature ($>800^\circ\text{C}$) of heating and a quick cooling rate the shearing of β lattice takes place, resulting in *HCP* α' phase, called non-diffusion martensite transformation in titanium alloys. So a sufficient heating temperature and a slower cooling rate are quite important for the gradient forming.

When the two-phase titanium alloys were treated with proper heating temperature and cooling rate, the gradient of alloying elements occurred, which also accompanied appearance of the gradient of electron concentration. The electron concentrations for Ti-5123 alloy were 3.927 in α , 3.954 in the interface phase, and 3.971 in β , respectively. And the shear modulus C' and the electron concentration are mutu-

ally dependant on each other in the alloys^[11]. During slow cooling of the titanium alloys, with the changing of electron concentration, their modulus C' goes down to minimum. Therefore, shearing, which generates easily on account of the misfitted volume, is able to relax the internal stress at the interface region.

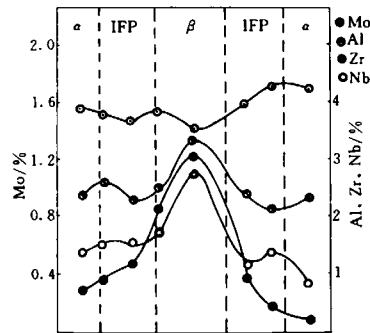


Fig. 7 EDX analysis of specimen aging at 800 °C

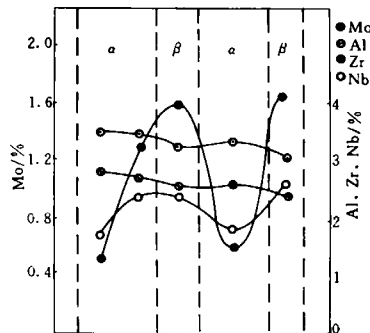


Fig. 8 EDX analysis of specimen aging at 700 °C

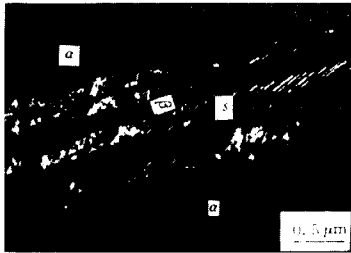


Fig. 6 Dark field of TEM from the interface phase $(\bar{1}101)_t$, aging at 800 °C/1 h

5 CONCLUSIONS

(1) There was not interface phase in the specimen after quenching and subsequently aging at below 800 °C. This means that the forming of the interface phase is related to heating temperature and cooling rate.

(2) There was interface phase in the specimen after treating at 950 °C for 1 h and water-quenching followed by aging at 800 °C. The interface phase had a double-layer structure with it. The monolithic layer was *fcc* and its orientation is $(001)_m // (110)_\beta$ and $[110]_m // [\bar{1}\bar{1}\bar{1}]_\beta$. The striated one was *HCP* with $(10\bar{1}1)\langle\bar{1}012\rangle$ twin relationship to α phase. Its thickness was about 100~300 nm.

(3) When the interface phase formed, the composition gradient at the interface occurred, which resulted in gradient of electron concentration. This probably is the main factor to create the interface phase in the titanium alloys. If no interface phase was formed, the concentration of alloying elements was discontinuous at α/β boundaries.

(4) The interface phase of titanium alloys

has nothing to do with the thinning method for TEM specimens and the so-called titanium hydride.

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