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X-ray quantitative phase analysis of super α_2 alloy through thermochemical processing with hydrogen

T. S. WANG, B. LIAO, F. R. XIAO
Department of Material Engineering, Yanshan University, Qinhuangdao 066004, People's Republic of China
K. YANG, Y. Y. LI
Institute of Metal Research, Academia Sinica, Shenyang 110015, People's Republic of China

The effect of hydrogen on the phase constitution of super α_2 alloy was studied by means of X-ray quantitative phase analysis. The results showed that the alloy consists of α_2, B_2 and O phases, and the volume fraction of O phase in the alloy increases with hydrogen content.

1. Introduction

Ti_3Al based alloy, a kind of high temperature structural material, will be in great prospect of application. However, its poor plasticity at room temperature limits its practical application. So researchers have tried to find some ways to enhance the room temperature plasticity of this alloy. Recently, procedures of thermomechanical processing [1–3] and thermochemical processing (TCP) with hydrogen [4–6] were used to modify the microstructure and room temperature properties of the alloy. In addition, some groups have observed O phase in Ti_3Al based alloys [7–9] and discovered that the alloys containing O phase have superior tensile strength, fracture toughness and creep resistance [10]. So that it is believed that the volume fraction of O phase in super α_2 alloy should play an important role in the improvement of room temperature properties. This paper studies the effect of hydrogen on phase constitution in detail, especially the volume fraction of O phase in super α_2 alloy by means of X-ray phase analysis to provide a theoretical instruction in improvement of the microstructure and properties of this alloy.

2. Experimental procedure

The material used in this study was a forged bar of super α_2 alloy of 20 × 20 mm size and had a nominal composition of Ti–25Al–10Nb–3V–1Mo (at %). The samples used in the experiments were pieces of 15 × 15 × 4 mm size. Hydrogenation of the samples was carried out at 800 °C for 0.5 h under three different hydrogen pressures, 0.05, 0.1 and 0.2 MPa, respectively. According to the results of thermal analysis, the hydrogen contents in the corresponding samples are 6, 10 and 14 at %.

Samples containing various contents of hydrogen were solutionized in the single phase β region and then aged in vacuum. The solutionizing temperature was chosen according to the effect of hydrogen contents on α_2/β transus temperature of super α_2 alloy [11]. The detailed processing procedures of the samples are indicated in Table I. The analysis of phase in the samples was carried out using an X-ray diffractometer (XRD) model D/MAX-rB, with CuK_α radiation and a graphite monochromator. The lattice constants used for phase analysis are shown in Table II. Energy dispersive spectrometry (EDX) was used to determine the composition of every phase in the samples.

2.1. Theoretical analysis

2.1.1. Calculation of structure factor

The structure factor F_{hkl} of the (hkl) crystal face can be calculated by the following formula

$$F_{hkl} = \sum_{j=1}^{n} \exp[2\pi(hx_j + ky_j + lz_j)i] \qquad (1)$$

where hkl is the crystal face index; x_j, y_j, z_j are position co-ordinates of atoms in the unit cell; and f_j is the scattering factor of atom j in the unit cell. Only three elements, Ti, Al and Nb, were considered in the calculation of the structure factor in this paper. From the crystal structures of α_2, B_2 and O phases (Fig. 1) and Equation 1, one can deduce the structure factors of the three phases, respectively.

2.1.2. Quantitative phase analysis

Using the same XRD and radiation in all the experiments, the diffraction intensity of the specified plane of every phase in the sample can be given by

$$I = K_0 N^2 F^2 P \phi(\theta) \frac{e^{-2M}}{2\mu_l} v \qquad (2)$$

where K_0 is a constant, N is the number of unit cell per unit volume, F is the structure factor, P is the

TABLE I Processing procedures of samples

Designation	Processing procedure[a]
A11	800 °C/0.5 h (V, FC) + 1180 °C/0.5 h (WQ) + 800 °C/2 h (V, FC)
B11	800 °C/0.5 h (0.05 MPa H_2, FC) + 1050 °C/0.5 h (WQ) + 800 °C/2 h (V, FC)
C11	800 °C/0.5 h (0.1 MPa H_2, FC) + 1000 °C/0.5 h (WQ) + 800 °C/2 h (V, FC)
D11	800 °C/0.5 h (0.2 MPa H_2, FC) + 970 °C/0.5 h (WQ) + 800 °C/2 h (V, FC)

[a] V, vaccum; FC, furnace cool; WQ, water quench.

TABLE II Lattice constants (nm) used

α_2 (ordered h.c.p.[a]) [12]	B_2 (ordered b.c.c.[b]) [13]	O (ordered orthorhombic [14]
$a = 0.578$	$a = 0.322$	$a = 0.608\,93$
		$b = 0.956\,94$
$c = 0.467$		$c = 0.466\,66$

[a] h.c.p., hexagonal close packed.
[b] b.c.c., body centred cubic.

multiplication factor, μ_l is a linear absorption factor, e^{-2M} is the Debye temperature factor, v is the volume of the phase to be determined, and $\phi(\theta)$ is the Lorentz polarization factor. Let V represent the volume of the sample. Then $f = v/V$ is the volume fraction of the phase to be determined. If there are little differences in the value of μ_l and e^{-2M} among the three phases, one has

$$\frac{I}{V} = KCf \tag{3}$$

where $K = K_0 e^{-2M}/2\mu_l$ and $C = N^2F^2P\phi(\theta)$. According to the analyses mentioned above, one can approximately consider K as a constant. If the sample consists of n phases, then

$$f_1 + f_2 + \cdots + f_n = 1 \tag{4}$$

From Equations 3 and 4, the volume fraction of every phase can be given by

$$f_i = \left(\frac{C_i}{I_i}\sum_{i=1}^{n}\frac{I_i}{C_i}\right)^{-1} \tag{5}$$

3. Results and discussion

Fig. 2 shows the XRD spectra of the samples with various hydrogen contents through solution and ageing treatment. The results showed that every sample consists of α_2, B_2 and O phases. The diffraction peaks $(2\,0\,3)\alpha_2$, $(1\,1\,0)B_2$ and $(2\,2\,0)_O$ are used for quantitative phase analysis in this paper, the corresponding structure factors can be given by the following equations

$$\begin{aligned} F^2_{(2\,0\,3)\alpha_2} &= 3(f_{Al} + 3f_{TiNb})^2 \\ F^2_{(1\,1\,0)B_2} &= (f_{AlNb} + f_{Ti})^2 \\ F^2_{(2\,2\,0)O} &= 4(f_{Ti} + f_{Al} + f_{Nb})^2 \end{aligned} \tag{6}$$

The EDX results of every phase are shown in Table III. It can be seen that the atom ratio of Ti to

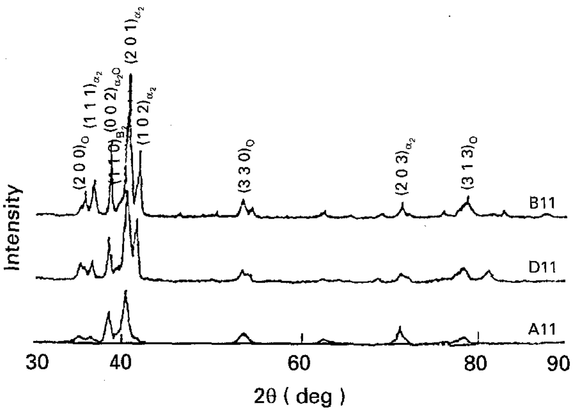

Figure 2 XRD spectra of the samples with solution and ageing treatment.

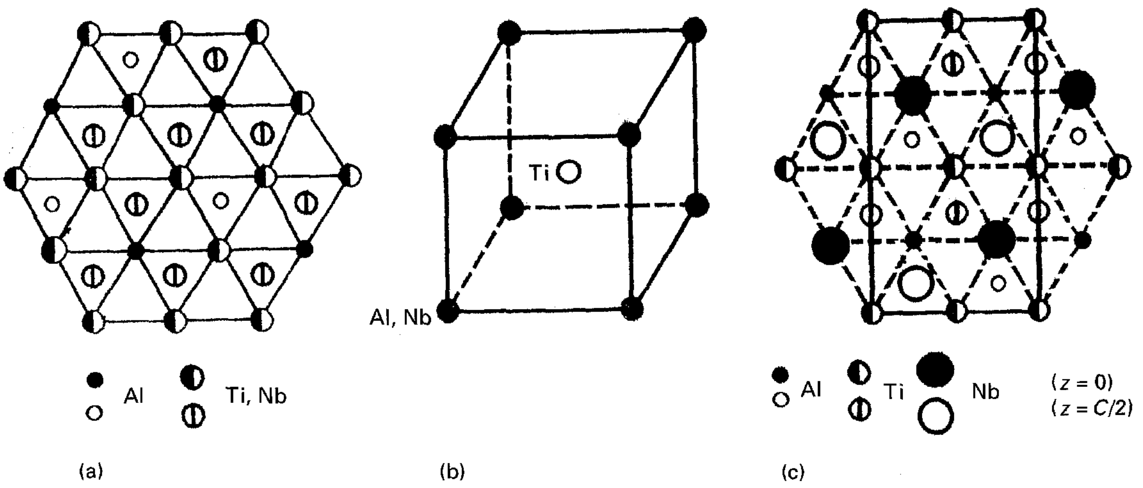

Figure 1 Sketches of α_2, B_2 and O structures: (a) [0 0 0 1] projection of α_2 structure [12], (b) structure of B_2 [13], and (c) [0 0 1] projection of O structure [12].

TABLE III EDX results of each phase studied (at %)

	α_2	B_2	O
Ti	59.95	46.38	51.17
Al	27.81	37.26	33.81
Nb	12.24	18.36	15.02

TABLE IV Parameters used for calculation and the calculated values of C

Parameter	$(2\,0\,3)_{\alpha_2}$	$(1\,1\,0)_{B_2}$	$(2\,2\,0)_O$
2θ, deg	71.36	39.58	34.93
F^2	6048.03	1018.89	20158.32
N, nm^{-3}	0.2467×10^{-3}	2.9953×10^{-3}	0.3677×10^{-3}
P	12	12	4
$\phi(\theta)$	3.914	13.67	17.89
C	1.729	149.95	19.51

TABLE V Integral intensity and volume fraction of α_2, B_2 and O phases

Sample	Integral intensity			Volume fraction (%)		
	$(2\,0\,3)_{\alpha_2}$	$(1\,1\,0)_{B_2}$	$(2\,2\,0)_O$	α_2	B_2	O
A11	297.13	86.46	82.64	97.33	0.33	2.34
B11	139.79	109.14	57.09	96.50	0.80	2.70
C11	458.83	530.83	251.33	94.17	1.26	4.57
D11	118.45	78.17	171.92	88.01	0.67	11.32

Nb in α_2 and Al to Nb in B_2 phases are 5:1 and 2:1, respectively. Thus one has

$$f_{\mathrm{TiNb}} = \frac{5}{6} f_{\mathrm{Ti}} + \frac{1}{6} f_{\mathrm{Nb}}$$

$$f_{\mathrm{AlNb}} = \frac{2}{3} f_{\mathrm{Al}} + \frac{1}{3} f_{\mathrm{Nb}} \qquad (7)$$

where f_{TiNb} is the weighted average for the scattering factors of atoms Ti and Nb in α_2 phase, and f_{AlNb} is the weighted average for the scattering factors of atoms Al and Nb in the B_2 phase. The maximum difference in μ_1 among α_2, B_2 and O phases is below 1.2% by the authors' calculation, and all the experiments were carried out at room temperature, therefore, the volume fraction of phase to be determined can be accurately calculated by Equation 5. Table IV shows the parameters used for calculation and the calculated values of C.

Separation of the peaks used for quantitative phase analysis mentioned above was carried out by means of the method proposed by Hindeleh [15], and the integral intensities of the corresponding peaks are listed in Table V. The volume fractions calculated by Equation 5 are also listed in Table V. Fig. 3 shows the relationship between the volume fraction of O phase and hydrogen content. The volume fraction of O phase increases with increment of hydrogen content, and increases obviously at 10 at % H. The volume fraction of O phase in the sample containing 14 at % H is about four times more than in the unhydrogenated sample. The variation of the volume fraction of α_2 phase is in contrast to that in O phase with hydrogen content.

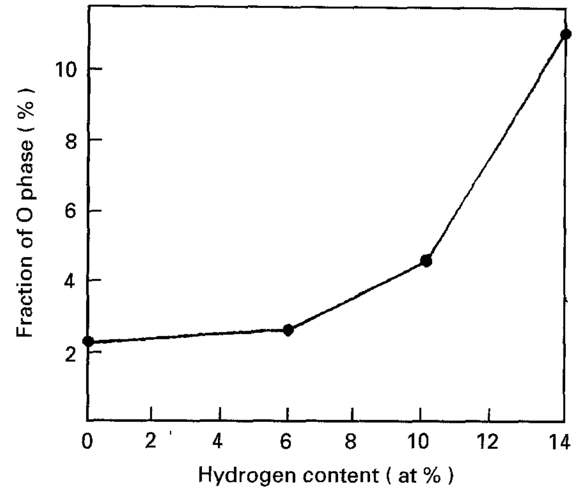

Figure 3 Relationship between the volume fraction of O phase and hydrogen content.

The variation of volume fraction of the phases in the samples with various hydrogen contents should be attributed to specific alloying effects of hydrogen in this alloy. Since hydrogen is a strong β stabilizer in the alloy, the behaviour of transformation $B_2 \rightarrow \alpha_2$ in the hydrogenated sample is very different from the unhydrogenated sample. First, hydrogenation lowers the α_2/β transus temperature and extends the incubation period of the isothermal transformation $B_2 \rightarrow \alpha_2$, which hinders the transformation $B_2 \rightarrow \alpha_2$. Second, because the compositional difference between O and B_2 phases is smaller than that between α_2 and B_2 phases, compositional modification from the B_2 phase to O phase is more easily achieved. In addition, the lattice distortion of B_2 phase caused by hydrogenation can have a positive effect on structural modification from the B_2 phase to the α_2 phase. Therefore, hydrogenation can obviously promote the formation of O phase in this alloy, which results in the increment of the volume fraction of O phase with hydrogen content.

4. Conclusions

The super α_2 alloy through thermochemical processing with hydrogen consists of α_2, B_2 and O phases. Hydrogenation obviously promotes the dynamic process of transformation of $B_2 \rightarrow O$, which leads to the increment of O phase in super α_2 alloy with hydrogen content.

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