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Phase relation in titanium–aluminide alloy – an X-ray study

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Results of X-ray diffraction studies on titanium aluminides stabilized by niobium, vanadium and molybdenum are reported to establish a phase relation in the Ti–25Al–10Nb–3V–1Mo at% (Ti–25–10–3–1) alloy. It is shown that the composition of the phases probably deviated slightly from ideal stoichiometry Ti_3Al for α_2 and Ti_2AlNb for β-type; its partial ordering in of the β-phase type and the phase relation is 64% β-type and 36% α_2 phase.

1. Introduction

Titanium aluminide intermetallics are being considered for use at high temperature because of their attractive high-temperature properties, and their improvements in ductility and fracture toughness achieved in recent years [1, 2]. The ordering of the β-phase to a B2 structure has been observed in several alloys: Ti–Al–Mo [3, 4], Ti–Al–V [5], and Ti–Al–Ta [5] systems.

This ordered structure appears to form around the composition Ti_2AlX [4, 5] where X is a β-stabilizing addition. In this study, X-ray diffraction was used to establish the phase relation in Ti–25Al–10Nb–3V–1Mo at % (Ti–25–10–3–1) alloy.

2. Experimental procedure

The studies were carried out on samples of Ti–25Al–10Nb-3V–1Mo at % alloy (Ti–25–10–3–1). The material was forged at 1900 °F (~ 1038 °C) and heat treated before and after being forged at 1985 °F (~ 1085 °C) for 1 h, and quenched in oil. The composition analysis of the alloy is indicated in Table I.

X-ray diffraction measurements were used to determine the amounts of α- and β-phases in this alloy and lattice parameters.

A conventional computerized Philips diffractometer with a copper anode tube and scintillation detector was used for diffraction study. The data were accumulated by step-scanning with 0.02°/steps and 4 s collecting time per step.

Calculation of the powder patterns for α_2- and β-phases was carried out using a Powder 12 computer program [6].

Samples for X-ray diffraction were cut by an electric spark erosion machine. The upper layer of the samples was removed by etching in HF solution.

3. Results

Two major phases were detected by X-ray diffraction: α_2- and β–Ti type. Experimental and calculated diffraction data for α_2- and β-phases are indicated in Tables II and III.

Lattice parameters of α_2 and β are shown in Tables IV and V.

α_2- and β-phases are ordered as follows from the appearance of super-lattice lines, 1 0 0 for β-phase and 1 0 1 for the α_2-phase. A comparison of the observed and calculated intensities showed a significant degree of preferred orientation in both phases. In the β-phase, the crystallographic plane 1 0 0 is oriented, preferentially parallel to the sample plane, and in the α_2-phase, the 0 0 1 plane has preferred orientation.

3.1. Quantitative analysis

The ratio of integrated intensity of two diffraction peaks, reflected by two phases (β and α_2 in our case) is given by Equation 1 [11]

$$\frac{(I_{hke})_\beta}{(I_{hke})_{\alpha_2}} = \frac{K_\beta}{K_\alpha}\frac{C_\beta}{C_{\alpha_2}} \qquad (1)$$

where C_β and C_α are the weight fractions of β and α_2 phases, K_β and K_α are calibration constants. The calibration constants are independent of composition and are defined by crystal symmetry, crystal structure and scattering angle. If the ratio K_β/K_α is known, Equation 1, together with the evident mass balance equation

$$C_\beta + C_{\alpha_2} = 1 \qquad (2)$$

can be solved for C_β and C_{α_2}. The constants K_β and K_{α_2} may be calculated from the known formula of structure crystallography, provided that positional and occupational parameters of the atoms in the unit cell are known.

It follows from Equation 1 that the ratio of constants $K_\beta/K_{\alpha_2} \equiv R_{\beta\alpha_2}$ is equal to the ratio of corresponding intensities for 1:1 composition of these two phases. Moreover, if any phase, W, is chosen as a reference and the values $R_{\beta W}$ and $R_{\alpha_2 W}$ are known then the

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TABLE I Chemical composition of Ti-25-10-3-1 alloy (wt %)

Ti	Al	Nb	V	Mo	O	N	Fe
bal.	13.9	19.10	3.1	1.79	0.066	0.005	0.060

TABLE II Observed and calculated diffraction data for α_2

h k l	d (nm)	I/I_0 observed (%)	I/I_0 calculated (%)
1 0 1	0.3410	7.8	5.4
2 0 0	0.2502	20.5	24.7
0 0 2	0.2328	43.5	26.2
2 0 1	0.2204	100	100
2 0 2	0.1704	18.3	13.5
2 2 0	0.1444	24	14.1

TABLE III Observed and calculated diffraction data for β-type phase

h k l	d (nm)	I/I_0 observed (%)	I/I_0 calculated (%)
1 0 0	0.3231	0.39	2.1
1 1 0	0.2287	100	100
2 0 0	0.1616	47.0	15.2
2 1 1	0.1319	34.0	27.2
2 2 0	0.1143	1.8	7.8
3 1 0	0.1023	14.0	11.2
3 2 1	0.0864	11.8	19.9

TABLE IV Comparison of lattice parameters of α_2 (in Ti-25-10-3-1) alloy from diffraction measurements

	This study	Ming Gao [7]	Ence and Margolin [8]	Blackburn [9]
a (nm)	0.578	0.578	0.576	0.5765
c (nm)	0.466	0.462	0.465	0.4625

TABLE V Lattice parameters of β-phase, Nb and pure β-Ti

	This study (β-type)	Nb	Pure β-Ti[a]
a (nm)	0.323	0.330	0.327

[a] Reduced to ambient temperature from the parameter observed at 1084 °C [10].

required coefficient, $R_{\beta_{\alpha_2}}$ is

$$R_{\beta_{\alpha_2}} = R_{\beta W}/R_{\alpha_2 W} \tag{3}$$

It is acceptable to choose corrundum as a reference phase W [12], and we used the calculated corundum ratios in our analysis. Corundum ratio was calculated together with relative intensities by the Powder 12 program [6], and was found equal to 10.32 for β and 5.29 for α_2.

As was noted in the previous section, β and α_2 phases exhibit preferred orientation. Thus straight application of Equation 1 to any pair of diffraction peaks might lead to erroneous results. So in order to overcome this drawback we followed the multi-peaks approach proposed by Dickson [13] for analysis of two-phase steel.

The ratio of the phase abundancies is expressed as

$$\frac{C_\beta}{C_{\alpha_2}} = \frac{R_{\alpha_2 C}}{R_{\beta C}}\left(\frac{1}{n_\beta}\sum_0^{n_\beta} I_\beta/I'_\beta \Bigg/ \frac{1}{n_{\alpha_2}}\sum_0^{n_{\alpha_2}} I_{\alpha_2}/I'_{\alpha_2}\right) \tag{4}$$

where $R_{\alpha_2 C}$ and $R_{\beta C}$ are calculated corundum ratios. I_β, I_{α_2} are observed integrated intensities of diffraction peaks of β and α_2 phases. I'_β, I'_{α_2} are relative calculated intensities of corresponding diffraction peaks (Tables III and IV). n_{α_2} and n_β are the number of observed peaks of α_2 and β phases. All peaks of α_2-phase and all peaks without 2 2 0 of β-phase were included in the calculation. Reflection 2 2 0 was not included because the information associated with this peak is the same as with the first-order reflection 1 1 0. The results of calculation by Equation 4 give $C_\alpha = 36\%$ and $C_\beta = 64\%$.

4. Discussion

X-ray diffraction showed that the α_2-phase has DO_{19}-type crystal structure with perfect ordering. The lattice parameters were determined to be $a = 0.578$ nm and $c = 0.466$ nm, which correspond well with those reported in the literature (see Table IV). So the composition of α_2-phase in Ti-25-10-3-1 may be regarded as Ti_3Al. The observed intensity of super-lattice line 1 0 1 in the α_2-phase (Table II) is close to the calculated intensity. The slight difference is explained by the preferred orientation that is found in this phase.

We compare the lattice parameter of β-type (Ti_2AlNb) with a lattice parameter of pure β-Ti that has the same B2 structure. The latter was reduced from the data available at 1084 °C by applying linear contraction to room temperature [10] (see Table V). The lattice parameter of the β-type phase is slightly lower (1.3%) than for the reduced parameter of β-Ti. We can explain this difference by partial substitution of titanium by aluminium which has a smaller atomic radius (0.143 nm for aluminium and 0.147 nm for titanium).

The intensity of the observed superlattice line 1 0 0 in the β-type phase is lower than the calculated data. We can suggest two reasons for this observation: incomplete ordering or variation in composition of β-type phase.

The intensity of the superlattice lines depends on the ratio of aluminium to niobium in the composition $Ti_2Al_{1-x}Nb_x$. The ratio of intensity 1 0 0 to regular 2 0 0 is shown in Fig. 1.

The observed intensity ratio I_{100}/I_{200} in our study is equal to 0.008 (see Table III). From Fig. 1 we can see that this ratio corresponds to the composition $Ti_2Al_{0.7}Nb_{0.3}$ ($x \cong 0.3$). However, elementary chemical calculations show that if the composition of β-type phase is $Ti_2Al_{0.7}Nb_{0.3}$, aluminium consumption

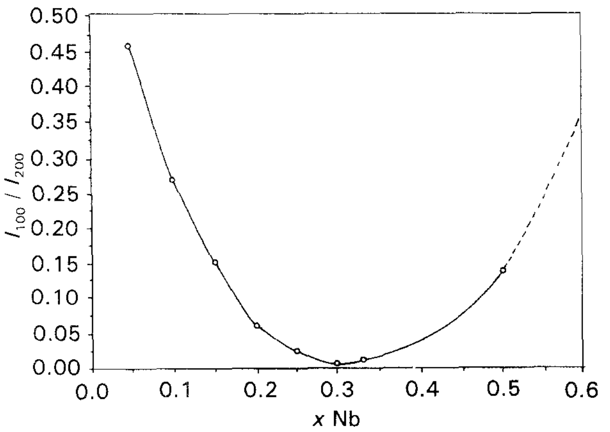

Figure 1 The calculated intensity of I_{100}/I_{200} for complete order.

in the β-type phase is 32 at %, and in our alloy (Ti–25Al–10 Nb–3V–1Mo) we have only 25 at % Al, so we can say that incomplete order is the main reason for the difference between the calculated and observed data of the superlattice line peak.

If we assume the ideal composition Ti_3Al for the α_2-phase and Ti_2AlNb for the β-type phase, and suppose that molybdenum and vanadium are equivalent to niobium, then chemical calculations show that the phase relation must be 56% for β and 44% for α_2. These data are not in complete agreement with X-ray analysis (64% β, and 36% α_2). This deviation is derived from the approximation of chemical composition of β-type phase. Probably the slight deviation from the ideal composition must be similar to $Ti_{2+x}AlNb$, as obtained by Banerjee *et al.* [14].

5. Conclusions

1. The Ti–25–10–3–1 alloy is composed of two phases: β-phase with B2 structure, and α_2 phase with DO_{19}-type crystal structure.
2. The phase relation is 64% β-type and 36% α_2-phase.
3. The composition of the phases probably deviates slightly from ideal stoichiometry Ti_3Al for α_2 and $Ti_2Al\,Nb$ for β-type.
4. Ordering is almost perfect in the α_2-phase, but only practical in the β-phase.

References

1. H. A. LIPSITT, in Proceedings of Materials Research Society Symposium 'High Temperature Ordered Intermetallic Alloys', edited by C. C. Koch, C. T. Liu and N. S. Stoloff Vol. 39 (1985) pp. 351–64.
2. N. S. STOLOFF, MRS, Pittsburg *ibid.* (1985) pp. 3–27.
3. H. BOHM and K. LOHBERG, *Z. Metallkde* **49** (1958) 173.
4. T. HAMAJIMA, G. LUTJERIZG and S. WEISMAN, *Met. Trans.* **3** (1972) 2805.
5. E. K. MOLCHENORA, "Phase Diagrams of Titanium Alloys", Israel Programme for Scientific Transformation, Jerusalem (1965) p. 167.
6. C. M. CLARK, D. K. SMITH and G. G. JOHNSON, a Fortran Program for Calculation of X-ray Powder Diffraction Patterns – Version 12. Department of Geo Science, Pennsylvania State University, University Park, PA, (1990).
7. MING GAO, J. BART and P. WEI, *Scripta Metall.* **2135** (1990) 24.
8. E. ENCE and H. MARGOLIN, *Trans. AIME* **221** (1961) 151.
9. M. BLACKBURN, *ibid.* **239** (1967) 1200.
10. J. SPREADBOROUGH and J. W. CHRISTIAN, *Proc. Phys. Soc. Lond.* **74** (1959) 609.
11. H. P. KLUG and L. E. ALEXANDER, "X-Ray Diffraction Procedures", 2nd Edn (Wiley, New York, 1974)
12. C. R. HUBBARD, E. H. EVANS and D. K. SMITH, *J. Appl. Crystallogr.* **9** (1976) 169.
13. M. J. DICKSON, *ibid.* **2** (1969) 76.
14. D. BANERJEE, T. K. NANDY and A. K. GOGIA, *Script. Metall.* **21** (1987) 597.

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