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Phase stability and mechanical properties of irradiated Ti–Al–V intermetallic compound

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Abstract

A Ti–35Al–10V intermetallic compound manufactured by powder metallurgy contains α_2, β and γ phases. It has a better strength and ductility than the Ti–Al binary alloy containing α_2 and γ phases. A typical 0.2% yield strength and total elongation of Ti–35Al–10V at 500 °C are 700 MPa and 15%, respectively. At 600 °C, the strength is still above 600 MPa and total elongation increases up to 60%. Transmission electron microscope (TEM) observation of the deformed microstructure suggests a transformation induced ductility in the β phase. After neutron irradiation of 3.5×10^{25} n/cm^2 at 400 and 600 °C, the total elongation is only 10% at the 600 °C test, and almost no plastic elongation was observed at the 400 °C test. The TEM observation of irradiated Ti–35Al–10V did not show the formation of the ω phase.

1. Introduction

Ti–Al intermetallic compounds are innovative high temperature materials with large strength-to-weight ratio and good oxidation resistance. Its density is comparable to that of ceramics such as SiC, and it has a good creep strength up to 1000 °C. Its elongation to rupture is much better than that of ceramics. These alloys are especially attractive in aerospace engineering and turbine parts. Advantages of Ti–Al intermetallic alloys are reflected to the conceptual design of the steady state Tokamak reactor (SSTR) [1], where the highest operating temperatures of structural material and He coolant are 900 and 700 °C, respectively. The contact dose rate of Ti–Al after ten-year cooling is 10^3 times lower than that of F82H, a typical reduced activation ferritic/martensitic steel, although the long life ^{26}Al whose half life time is 7.2×10^5 years makes the decrease of activity very slow after that [2].

A major problem for these materials to be used in structural components is their low ductility at relatively low temperatures. Efforts have been devoted to improve their ductility by microstructural control. Effects of third elements have been also examined and recent results show that the addition of vanadium more than 10 at.% is very effective to improve their ductility [3–5]. A Ti–40Al–10V alloy showed a total elongation of 6% at ambient temperature test [4]. In these alloys, the microstructure consists of three phases, α_2, γ and β with B2 structure (CsCl structure). A Ti–35Al–15V showed a transformation-induced ductility above 620 °C leaving a characteristic deformation structure in β grains [5]. This suggests an instability of β phase in deformation at elevated temperature. The stability of β phase under irradiation and its effect on mechanical properties are also of great concern. In this work, a Ti–35Al–10V alloy, also including β phase, was neutron-irradiated. Tensile tests and electron diffraction analysis using a transmission electron microscope (TEM) were performed.

2. Experimental procedure

The Ti–35Al–10V powder was prepared by plasma rotating electrode process (PREP) [6]. The consuming electrodes for PREP were prepared by two-step vacuum melting. The chemical compositions of the electrode were 36.0 at.% Al, 10.7 at.% V, 0.1 at.% O. An

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evacuated can of Type 304 containing the powder was hot-isostatic-pressing (HIP) treated at 1160 °C and 1800 bar for 3 h, followed by an isothermal-hot-forging (IHF) process at 950 °C. The height of the can was reduced by 80% in the IHF process with the initial strain rate of 1.1×10^{-4} s^{-1}.

Small flat tensile specimens were obtained from the IHFed pancake-shaped product. The gauge section of this type of specimen is 7.62 mm long, 1.52 mm wide and 0.76 mm thick. The surface of the gauge section was finally polished by #1200 or finer emery paper. Specimens were neutron irradiated in the Japan Research Reactor No. 3 Modified (JRR-3M) at 400 and 600 °C to the fluence of 3.5×10^{25} n/cm^2. Some specimens were thermally aged in evacuated quartz tubes.

Two tensile test machines were used, one in the hot cell and the other in a cold area. Both cold and hot tests were conducted with a strain rate of 1.1×10^{-4} s^{-1} in a vacuum. Thermally aged specimens were ruptured by the cold test machine.

TEM specimens were obtained from IHFed pancake, deformed tensile specimens (cold test) and 3 mm disks neutron-irradiated or thermally aged. Mechanically polished 3 mm disks obtained from the as IHFed product were also used for scanning electron microscopy (SEM). Details of electrolytical thinning of cold specimens are described elsewhere [4]. Foil specimens from neutron-irradiated disks were obtained using a focused ion beam (FIB), FB-2000A with a micro-sampling system.

3. Results and discussion

3.1. Ti–35Al–10V as manufactured

Results of tensile tests of as IHFed Ti–35Al–10V are summarized in Fig. 1 where those of binary TiAl alloy (Ti–47Al) manufactured by similar method are also included [3,7]. As shown in Fig. 1(a), the 0.2% yield strength of ternary Ti–35Al–10V is more than 600 MPa up to 600 °C and is better than that of Ti–47Al in this temperature range. Fig. 1(b) shows that the Ti–35Al–10V has better ductility than the binary alloy at intermediate temperature and above. In particular, the total elongation reaches 60% at 600 °C.

On the other hand, the ductility of Ti–35Al–10V at ambient temperature is low, 1%. Another β-containing alloy (Ti–40Al–10V) showed a total elongation of 6% at ambient temperature [4], while these two alloys have similar ductility at 600 °C. These two alloys are, however, manufactured differently. Present Ti–35Al–10V is manufactured through powder metallurgy, while the Ti–40Al–10V is prepared from a vacuum-melted ingot. Although HIP and IHF processes are employed in manufacturing both alloys, SEM observation revealed small cracks of several microns on polished surface of both alloys. The number of these defects was much fewer in Ti–40Al–10V than Ti–35Al–10V. It is highly possible that the ductility of Ti–35Al–10V at ambient

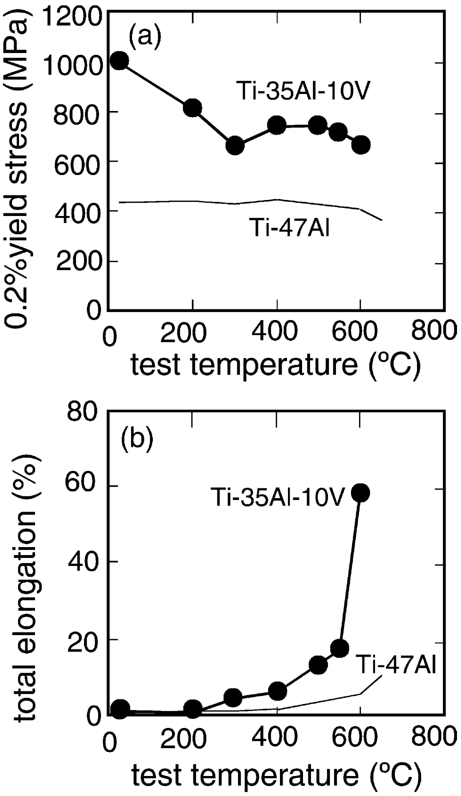

Fig. 1. Results of tensile tests of as IHFed Ti–35Al–10V. Results of a binary alloy (Ti–47Al [3,7]) manufactured by similar method are also included for comparison.

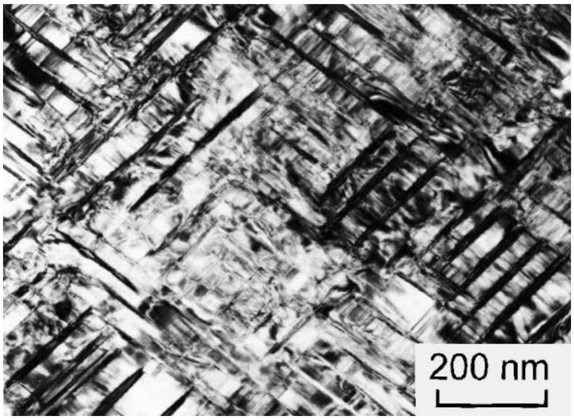

Fig. 2. A β-phase grain of Ti–35Al–10V deformed at 600 °C.

Table 1
Tensile test results of irradiated specimens

	Irradiation condition					
	Unirradiated		400 °C		600 °C	
400 °C test temperature						
ID	TVL	TVN	TVB	TVC	TVI	TVJ
0.2% yield stress (MPa)	769	784	N.A.	N.A.	N.A	N.A.
Maximum stress (MPa)	991	1190	1250	735	688	774
Uniform elongation (%)	2.4	14	0	0	0	0
Total elongation (%)	2.4	14	0	0	0	0
600 °C tests temperature						
ID	TVM	TVO	TVD	TVE	TVF	TVH
0.2% yield stress (MPa)	675	664	774	774	799	784
Maximum stress (MPa)	1051	1052	1024	1052	1021	999
Uniform elongation (%)	16.2	15.8	6.2	6.4	10.0	7.4
Total elongation (%)	58.5	75.1	6.2	6.4	10.0	7.4

temperature would be improved by reducing these small defects.

An abrupt increase of rupture elongation around 600 °C has been also observed in other β-containing Ti–Al–V alloys [3–5], and a characteristic deformation structure in β-phase grains is reported in a Ti–35Al–15V alloy [5]. Present Ti–35Al–10V alloy has a microstructure of equiaxial and fine grains (one to a few microns in diameter) [3]. The volume fraction of β phase is more than 50%, although an accurate measurement was not made. Fig. 2 shows a β-phase grain of Ti–35Al–10V deformed at 600 °C. The reported deformation structure in Ti–35Al–15V includes transformation bands of hexagonal α_2 grains and β grains in two different orientations [5]. Their orientation relationships $(101)\beta//(0\,0\,0\,1)\alpha_2$ and $[1\,1\,1]\beta//[1\,2\,1\,0]\alpha_2$ suggest martensitic transformation of β phase. In the microstructure shown in Fig. 2, the individual transformation bands reported for Ti–35Al–15V were not clearly distinguished, but the diffraction analysis suggests the formation of hexagonal phase. It should be noted that the reported deformation structure in Ti–35Al–15V included only one system based on a specific matrix $(1\,0\,1)\beta$. Although the formation mechanism of transformation bands is not clarified yet, Fig. 2 shows more complicated microstructure, probably including more than two systems on matrix $\{1\,0\,1\}\beta$.

3.2. Irradiated and thermally aged Ti–35Al–10V

Tensile results of irradiated specimens obtained with the tensile test machine in the cell are summarized in Table 1. Results on unirradiated specimens obtained with the same machine are also included, showing a good agreement of 'cold' and 'hot' tensile test machines. At 400 °C tests, specimens irradiated at 400 and 600 °C showed almost no plastic deformation before fracture. The 0.2% yield stress of irradiated specimens was thus not available. At 600 °C tests, irradiated specimens showed plastic deformation before fracture but the total elongations were much smaller than those of unirradiated specimens. One specimen, ID TVE, irradiated at 400 °C was kept at 600 °C for 6 h before the tensile test at 600 °C, but the total elongation was only 6.4%. The ductility of present Ti–35Al–10V tested at 400 and 600 °C was highly reduced by the neutron irradiation. Tensile results of thermally aged specimens ruptured at 600 °C are summarized in Table 2. The 0.2% yield stress

Table 2
Tensile results of thermally aged specimens tested at 600 °C

ID	62	7A	6	3
Aging condition				
Temperature (°C)	600	700	900	1100
Time	40 days	18 days	80 h	3 h
0.2% yield stress (MPa)	759	740	714	773
Uniform elongation (%)	13.7	14.2	17.2	0.9
Total elongation (%)	48.2	60.3	64.9	3.8

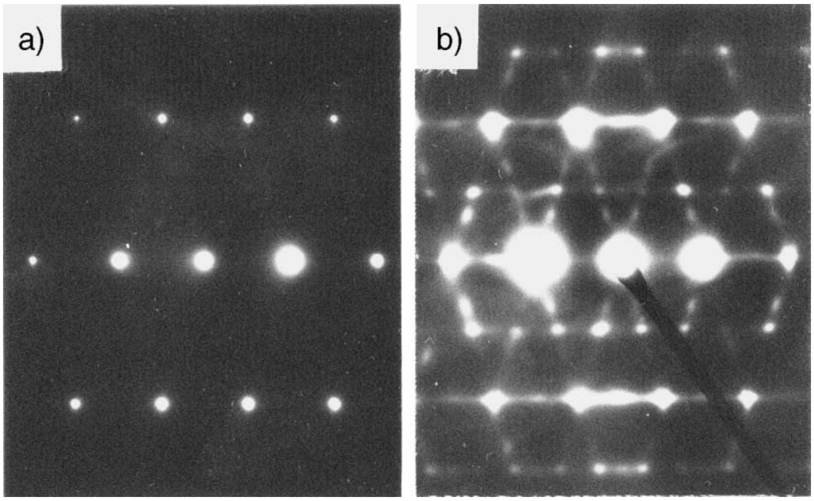

Fig. 3. Electron diffraction patterns obtained from β phase grains in 600 °C-irradiated (a) and 1100 °C-aged (b) specimens. Both are obtained with similar orientation, B ~ [1 1 3]. Extra reflections in (b) are from ω phase [8].

changes very little in these aging conditions. The total elongation keeps the same level, about 60%, or slightly increases as the aging temper increases up to 900 °C. These data are consistent with the elongation value of the as IHFed specimen tested at 600 °C (Fig. 1). But the total elongation suddenly decreases down to 3.8% at 1100 °C aging, although the 0.2% yield stress shows no remarkable change.

Fig. 3(a) and (b) show electron diffraction patterns from β phase grains in 600 °C-irradiated and 1100 °C-aged specimens, respectively. The ductility was highly reduced after these treatments. Both diffraction patterns are obtained with similar orientation, with the beam direction B close to [1 1 3]β. The electron diffraction pattern of 1100 °C-aged specimen (Fig. 3(b)) includes additional reflections, which are hardly observed in that of irradiated specimen (Fig. 3(a)). The characteristics of additional reflections in Fig. 3(b), the position and the direction of streaking, are quite similar to that of ω phase formed in a Ti–35Al–15V alloy [8]. It can be thus concluded that the embrittlement of 1100 °C-aged specimen is caused by the formation of ω phase. On the other hand, formation of ω phase due to irradiation was not detected. Marked reduction of ductility due to irradiation, especially the poor ductility of 400 °C-irradiated and 600 °C-tested specimens may suggest that the embrittlement is caused by phase decomposition in addition to the hardening due to radiation defects. But it was not the case.

4. Summary

A Ti–35Al–10V intermetallic compound containing α_2, β and γ phases was fabricated with powder metallurgy. It has better strength and ductility than a Ti–Al binary alloy manufactured by a similar method but its ductility at ambient temperature has not reached to that of a Ti–40Al–10V fabricated from a casted ingot. Neutron irradiation at 400 °C and 600 °C caused marked degradation in ductility but the electron diffraction analysis did not show the formation of ω phase in irradiated β grains, which was detected 1100 °C-aged material and has caused marked degradation in ductility.

References

[1] S. Mori et al., Fus. Technol. 21 (1992) 1744.
[2] Y. Seki et al., J. Nucl. Mater. 258–263 (1998) 1791.
[3] A. Hishimuma et al., Phys. Stat. Solid (a) 167 (1998) 521.
[4] A. Hishinuma, M. Tabuchi, T. Sawai, Intermetallics 7 (1999) 875.
[5] T. Sawai et al., Mater. Res. Soc. Symp. Proc. 650 (2000) R3.
[6] M. Tokizane, R. Kumagaya, International Conference on PM Aerospace Maerial 1991, 4–6 November 1991, Lousanne, Switzerland, paper 26.
[7] A. Hishinuma et al., Intermetallics 4 (1996) 179.
[8] G. Shano, A.P. Miodownik, T. Tsakiropoulos, Philos. Mag. 71 (1995) 1389.