CHAPTER 3 EXPERIMENTAL PROCEDURE

3.1 Introduction

This work was divided into two major parts. The first part was to develop a technique for the synthesis of titanium aluminides from titanium scrap and aluminum ingot available locally. The properties and structures of the synthesized titanium aluminides were then investigated. Ingot of titanium aluminides, weighing approximately 60 g, were produced by melting in an arc melting furnace with a non-consumable electrode and cast into a water-cooled copper mold in a pure argon (99.995%) atmosphere. The alloys produced were Ti-46 at.%Al and Ti-48 at.%Al with minor elemental additions of niobium (approximately 5 at.%Nb and 10 at.%Nb). The as-cast microstructures, crystal structures and microhardness values were examined and tested. The second part of this work focused on the effects of alloying elements and heat treatment process (solution treatment) as well as various cooling rates (cooled by water, oil, air, and furnace cooled) on the properties, microstructures, crystal structures, and phase orientations. The compositions of the alloys were synthesized with Nb as a ternary element and Mo and Cr as quaternary elements as follows: Ti-46Al, Ti-46Al-4Nb, Ti-46Al-2Mo, Ti-46Al-2Cr, Ti-46Al-4Nb-2Mo, and Ti-46Al-4Nb-2Cr.

(a) Ti 99.5% purity

(b) Al 99.80% purity

(c) Nb 99.80% purity

(d) Mo 99.95% purity

Figure 3.1 Starting materials used in this work

3.2 Synthesis of titanium aluminides

3.2.1 Materials

The TiAl-based alloys were synthesized from the following high purity raw materials: Ti (99.5%), Nb (99.80%) , Mo (99.95%) and Al (99.80%), as shown in Figure 3.1. Chromium was used in powder form, with a mesh measurement of approximately -60, 99.99% from Alfa Aesar, a Johnson Matthey Company. The chemical compositions of

the raw materials were verified before the melting process. A BAIRD DV6 Optical emission spectrometer was used to analyze the aluminum scrap, and the energy dispersive X-ray spectrometry (EDS) technique (as part of a Hitachi S-3400N scanning electron microscope) was used to analyze the titanium, niobium, molybdenum, and chromium. The results of the chemical composition analyses are shown in Tables 3.1-3.4

Table 3.1 Chemical composition of the Nb 99.80 at.% purity.

Nb	Fe	Si	Ni	W	Ti
balanced	0.004	0.003	0.002	0.004	0.002
Ta	O	C	H	N	-
0.071	0.015	0.004	0.0015	0.002	-

* From Alfa Aesar, a Johnson Matthey Company

Table 3.2 Chemical composition of the Mo 99.95 at.% purity.

Mo	Fe	Ni	Al	Si	Mg	C	N	O
balanced	0.01	0.005	0.002	0.01	0.002	0.01	0.003	0.008

* From Alfa Aesar, a Johnson Matthey Company

Table 3.3 Chemical composition of the Al 99.80 wt.% purity.

Al	Si	Fe	Cu	Ca	Mg	Zn	Mn
balanced	0.0495	0.0865	0.0039	0.0019	0.0198	0.0268	0.0152

* From BAIRD DV6 Optical emission spectrometer

Table 3.4 Chemical composition of the Ti 99.50% at. purity.

Ti	N	C	H	Fe	O
balanced	0.03	0.11	0.02	0.21	0.18

* From EDS analysis

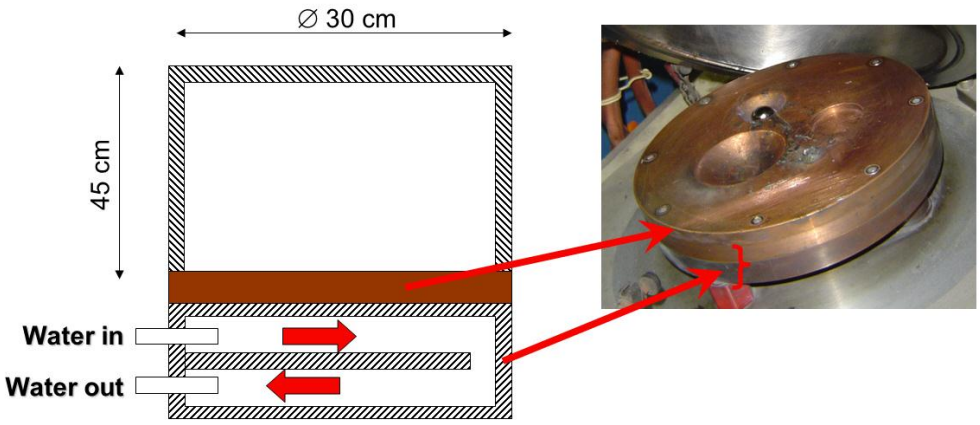

Figure 3.2 Water-cooled copper hearth of the arc melting water-cooled furnace

Figure 3.3 High-temperature heat treatment furnace

3.2.2 Charge calculation

Alloy compositions were determined based on the chemical composition expressed in atomic weight. The nominal composition, i.e., Ti-46Al-4Nb-2Mo, for 60 g can be calculated as follows:

Table 3.5 Charge calculation

elements	at.% (g/mol)	ratio	at.%×ratio	wt.% for charge (g)
Ti	48.88	48 at.%	2346.24	33.95*
Al	26.89	46 at.%	1236.94	17.90
Nb	92.91	4 at.%	371.64	5.38
Mo	95.94	2 at.%	191.88	2.78
Total	264.62	100 at.%	4146.70	60

* Calculate from: (2346.24/4146.70)×60 = 33.95 g

3.2.3 Melting method

The starting materials were melted into a button-shaped ingot with weights of approximately 60 g using an arc melting furnace with a non-consumable tungsten electrode under high purity argon (99.995%) atmosphere in a water-cooled copper hearth (Figure 3.2). The ingots were re-melted 5-6 times to ensure chemical homogeneity.

The experimental alloys are classified into two groups. The first group of alloys were; Ti-46Al, Ti48Al, Ti-46Al-5Nb, Ti-48Al-5Nb, Ti-46Al-10Nb, and Ti-48Al-10Nb (at.%). The second group of alloys were; Ti-46Al-2Mo, Ti-46Al-2Cr, Ti46-4Nb-2Mo, and Ti-46Al-4Nb-2Cr.

The energy dispersive X-ray spectrometry (EDS) technique (as a capability of a Hitachi S-3400N scanning electron microscope) was used to analyze the chemical compositions of the melted alloys, and the results are shown in Table 3.6

Table 3.6 Chemical compositions of experimental alloys

Alloys	Ti	Al	Nb	Cr	Mo
Ti-46Al	Balance	45.86	-	-	-
Ti-48Al	Balance	47.96	-	-	-
Ti-46Al-5Nb	Balance	45.91	4.63	-	-
Ti-48Al-5Nb	Balance	47.84	4.86	-	-
Ti-46Al-10Nb	Balance	45.83	9.91	-	-
Ti-48Al-10Nb	Balance	47.68	9.77	-	-
Ti-46Al-2Mo	Balance	45.96	-	-	1.91
Ti-46Al-2Cr	Balance	45.88	-	1.88	-
Ti-46Al-4Nb-2Cr	Balance	45.78	3.81	1.78	-
Ti-46Al-4Nb-2Mo	Balance	45.80	3.79	-	1.86

3.3 Heat treatment process

The as-cast button ingots were heated in the high temperature furnace at a temperature of 1,400°C (corresponding to the single-phase area) under argon atmospheres for 30 min and then subsequently air-cooled (AC). The ingots from the solution treatment were them cut into cubic specimens with nominal dimensions of 10 mm ×10 mm ×10 mm. All specimens were reheated in the high temperature furnace at a temperature at 1,350°C for 60 min. Then, the specimens were cooled either by oil quenching (OQ),

water quenching (WQ), air cooling (AC), or furnace cooling (FC), as shown in Figure 3.4.

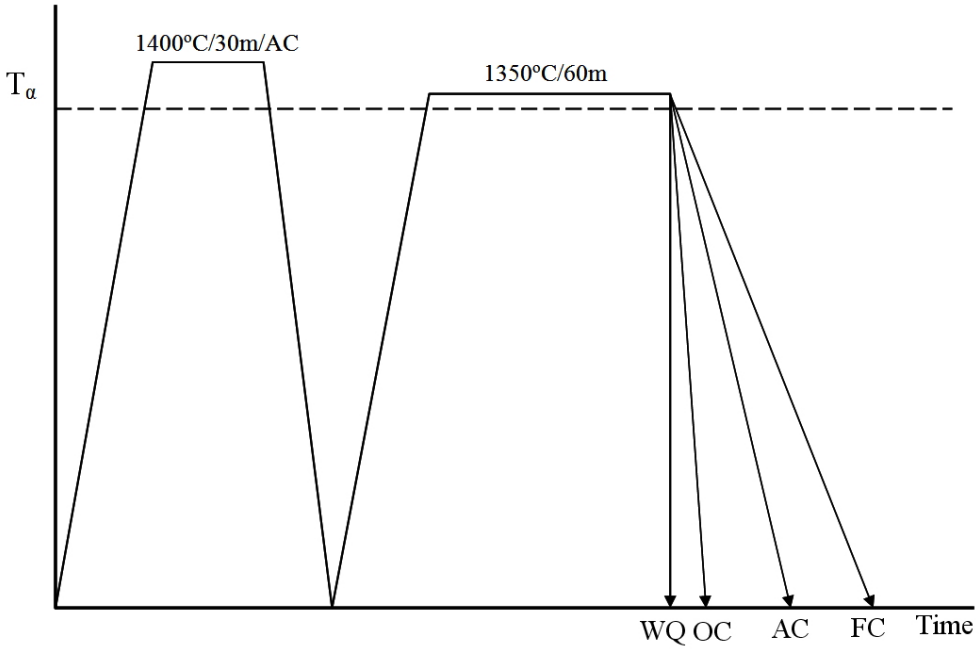

Figure 3.4 Schematic chart of the heat treatment process

3.4 Macrostructure and microstructure observations

3.4.1 Sample preparation and structural analyses

The as-cast and heat-treated specimens were abraded with SiC-based emery papers to remove the oxidized surface layers. Then, the specimens were rinsed with water, polished to a 0.1-μm finish with a diamond suspension abrasive, and etched using Kroll's regent (5 mL HF, 10 mL HNO_3, and 85 mL H_2O). Macrostructures of the specimens were studied using a Leica Z40 optical microscope. Microstructural observations were carried out using a Carl Zeiss optical microscope with a Leica Q-Phase image analyzer.

3.4.2 SEM and EBSD analyses

The heat-treated specimens, following polishing to a 0.1-µm finish with a diamond suspension abrasive, were polished by vibrational polishing process using a Buehler VibroMet Vibratory Polisher for 8 hrs. The specimens were then etched with Kroll's regent (5 mL HF, 10 mL HNO_3, and 85 mL H_2O). SEM and EBSD analyses were investigated using a Hitachi S-3400N scanning electron microscope. For detailed components of the phases, the back-scattered electron detection (BSE) mode was employed. For phase orientation with Kikuchi maps and pole figures, the electron back-scatter diffraction (EBSD) techniques was used. For chemical composition mapping, the energy dispersive X-ray spectrometry (EDS) was employed.

3.4.3 X-ray Diffraction

The specimens were abraded with SiC-based emery paper to remove the oxidized surface layer, rinsed with water, and then polished to a 0.1-µm finish with a diamond suspension abrasive. The crystal structures were analyzed using a Bruker D8 Advance X-ray diffractometer (XRD).

3.5 Microhardness test

The specimens were abraded with SiC-based emery paper to remove the oxidized surface layer, rinsed with water, and then polished to a 0.1-µm finish with a diamond suspension abrasive. The microhardness tests were performed using a MATSUZAWA Model: MMT-X7B hardness tester with 1,000 gf.