PREPARATION OF ALLOYS BASED ON INTERMETALLIC COMPOUNDS BY VIM WITH CENTRIFUGAL CASTING

SZURMAN Ivo[1], ČEGAN Tomáš[1,2], KURSA Miroslav[1]

[1] *VSB - Technical University of Ostrava, Ostrava, Czech Republic, EU*
[2] *Institute of Materials and Machine Mechanics, Slovak Academy of Sciences, Bratislava, Slovakia, EU*

Abstract

Intermetallic compounds based on Ti-Al, Ti-Ni, Fe-Al, Ni-Al and other systems require specific conditions during melting in a high-frequency vacuum induction furnace, especially material for melting crucibles and batch arrangement. The technology of casting plays an important role, too. The most common technology, very often used, is gravity casting. However, this method has certain disadvantages, namely frequent possibility of occurrence of cavities in the upper parts of the castings. Another possibility, currently often used, is technology of centrifugal casting. In this case several variants of arrangement of the device exist. With the combined vacuum-induction-centrifugal casting systems it is possible to realize the precision fine casting very economically with a compact system, also under vacuum and/or protective gas. As a melting crucible, oxide ceramic materials based are very often used, namely Al_2O_3, CaO, ZrO_2, with addition of Cr_2O_3 and others. Next material, which is very often used for manufacturing of melting crucibles, is graphite of various quality. This article deals with examination of melting and centrifugal casting of Ti-Ni and Ti-Al-X alloys with use of graphite crucibles. Both types of alloys are very sensitive to carbon content. It is known, that alloys based on titanium are during melting very reactive with graphite crucible, so the carbon content determination was realized in all prepared alloys. Microstructures were evaluated by optical microscopy (OM) and SEM techniques. For fabrication of experimental material casting mould with the inner diameter of 20 mm and length of 225 mm was used.

Keywords: Vacuum induction melting, centrifugal casting, Ti-Al alloy, Ti-Ni alloy

1. INTRODUCTION

The alloys based on intermetallic compounds TiNi and TiAl are used in technical practice for many years already. Technologies of their preparation are currently also considerably optimised. At present especially vacuum induction melting in ceramic [1] and graphite crucible [2, 3] of higher qualities (Ti-Ni iTi-Al), as well as ISM procedures, particularly for Ti-Al [4], are currently used as key technologies for preparation of these materials. As it is well known, a martensitic transformation takes place in an TiNi intermetallic compound. Properties of materials based on Ti-Ni-X depend, in comparison with classical materials, much more on chemical composition, structure and thermal-mechanical treatment. Even very small deviation from composition is sufficient for changing the transformation temperature M_s (martensite start) by several °C. For example change of the Ni content by 0.1 at.% causes change of the temperature M_s by 10 - 15 °C. By changing the alloy composition within the interval from 49 to 51 at.% Ni it is thus possible to change the temperature M_s from -200 °C to +120 °C [5]. This dependence may be on the one hand a negative feature of material, from the perspective of reproducibility of experimental results, on the other hand it is favourable from the viewpoint of its influence on thermal stability of austenitic and martensitic phase in dependence on chemical composition and microstructure. Transformation temperature $B2 \rightarrow B19'$ can be significantly reduced for example by alloying of equiatomic TiNi with iron or copper, when Fe and Cu atoms substitute the Ni atoms. This means that the ordered phase B2 [6] gets stabilised at the same time. Alloying with Cu or Fe may also reduce the concentration dependence of transformation temperatures and influence favourably mechanical properties [7]. Shape memory behaviour is new specific type of properties of technical alloys, which find extensive applications both as substitutes of conventional approaches, as well as in projects of completely new functional components. SMA materials also dump vibration very well, that provides a unique set of properties. It offers various multiple possibilities of applications, such as thermostatic switches, fire-fighting automatic circuit breakers, flexible antennas for mobile phones, air-conditioning regulators, active endoscopes in medical science, control elements in electrical engineering, physical measuring instruments, combat aeroplanes, micro-drives, automotive industry, solar technology, etc.

Ti-Al based alloys represent a unique group of materials with comparatively low density (3.8 - 4.2 g.cm^{-3}), high modulus of elasticity and wide temperature interval of usability. They show moreover for example good resistance to creep [8]. Such properties predetermine use of those alloys at the temperatures up to 800 °C, where they offer a possibility of their use as substitute of the existing Ni superalloys. However, at present a broader spectre of applications of these alloys is considerably limited by difficult methods of their preparation, by high prices of the prepared alloys, as well as not quite satisfactory formability under room temperatures.

2. EXPERIMENTAL

The medium-frequency melting and casting equipment “Supercast-Titan” was used for melting. From the Ti-Ni system was chosen the alloy Ti-50.85Ni (at.%), and from the system Ti-Al system the alloy Ti-47Al-8Ta (at.%). The used equipment has been designed preferably from vacuum induction melting and casting of Ti-alloys and γ-TiAl intermetallic compounds. This equipment represents in combination with vacuum induction melting and with centrifugal casting a compact unit. It is possible to work in vacuum or under atmosphere of inert gas (argon, nitrogen, mixed gas, etc.). This type of equipment makes it possible to prepare without bigger complications also other types of alloys -Co-Cr system, steel, stainless steel, Fe-Al alloys, Cu alloys, Ni alloys and Ni-Al, Ti-Ni based materials, as well as many others. It is also possible to apply method of investment casting. **Fig. 1** [9] shows scheme of the equipment. The following materials were used for preparation of alloys: Ni - 99.99%, Ti - 99.99%, Al - 99.9%, Ta - 99.9%. Isostatically pressed graphite crucible with inside diameter of 66 mm and height of 135 mm was used for preparation of experimental material. After setting and rotation of the casting arm the melt was cast into a graphite mould. In this way a casting with diameter of 20 mm and length of 225 mm was cast. **Fig. 2** shows prepared alloys.

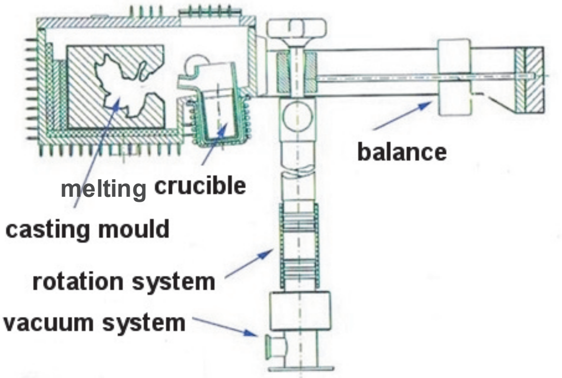

Fig. 1 Scheme of the Linn Supercast-Titan device [9]

Fig. 2 Experimental alloys after VIM a centrifugal casting

2.1. Characterisation of as-cast alloys

After melting the prepared alloys were assessed from the perspective of observance of the required chemical composition by OM and SEM methods. Samples for metallographic observations were taken from the volume of materials by classical methods. Moreover metallographic polished sections were prepared with use of a SiC abrasive material (grain fineness 80 - 2000) with subsequent polishing under water suspension of Al_2O_3 with particles size of 1 and 0.3 µm. Classical metallography was performed on the inverse metallographic microscope Olympus GX51 with camera DP12 and image processing system AnalSIS five.

SEM observations were performed by scanning electron microscopy in the mode of back-scattered electrons (BSE) and secondary electrons (SEM) using microscope QUANTA FEG 450, equipped with an energy dispersive spectrometer EDAX (EDS). EDS analysis was performed on two transversal sections taken from the bottom and upper part (below the feeding head) of each cast bar.

Average chemical composition of present phases of the bar was calculated from three independent measurements. Samples for measurements of oxygen and carbon content with dimensions of 5x5x5 mm were cut from the as-cast bars and wet grinded on SiC papers with grain size of 600. Oxygen content was measured by thermo-evolution method by analyser ELTRA ONH-2000. The measured sample was placed into a graphite crucible and heated in a resistance furnace under flowing helium. During melting oxygen reacts with the graphite, and oxygen content in the sample was analysed in the form of CO and CO_2 by infrared detector. Carbon content was measured by thermo-evolution method by analyser ELTRA CS-2000. The measured sample was placed in a ceramic crucible and inductively heated with tungsten under flowing oxygen. Tungsten serves as burning accelerator and carbon content is measured in the form CO and CO_2 by infrared detector. Average content of oxygen and carbon in each as-cast bar was calculated from three independent measurements.

3. RESULTS AND DISCUSSION

3.1. Ti-47Al-8Ta alloy

The alloy was prepared from elementary materials and master alloy Ta-Al, described in [10] with the size of bits of input materials up to 30x15x10 mm. The crucible before the melting was not exposed to melting for creation of TiC layer on its surface - as it is recommended by some authors in the case of Ti-Ni-X alloys. **Fig. 3** [3] shows microstructure of the alloy Ti-47Al-8Ta in the as-cast condition.

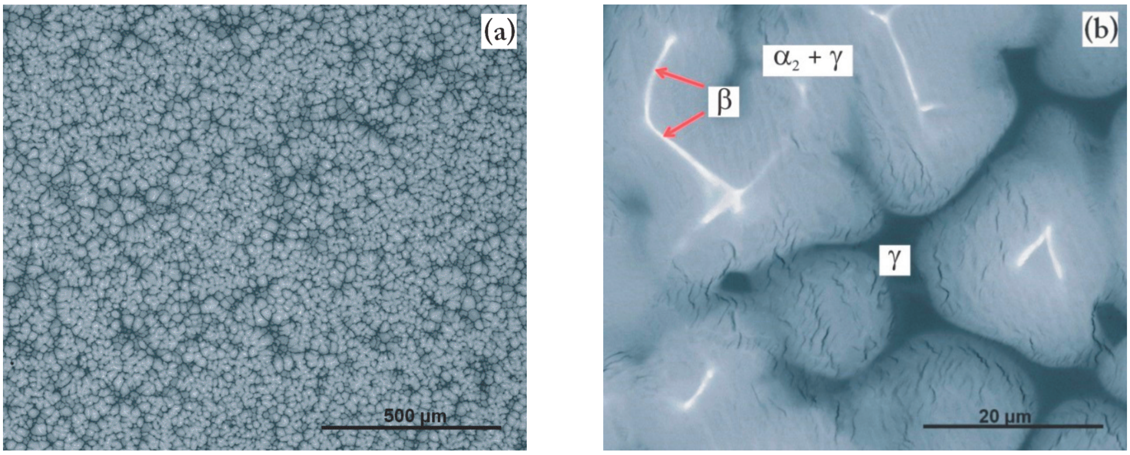

Fig. 3 BSE micrographs showing microstructure of as-cast Ti-47Al-8Ta alloy: (a) dendritic microstructure; (b) remaining β phase within the dendrites, lamellar $\alpha_2 + \gamma$ microstructure and interdendritic γ phase [3]

Typical dendritic micro-structure is illustrated in detail in **Fig. 3b**. After centrifugal casting the microstructure was characterised as lamellar $\alpha_2 + \gamma$ with residual particles of the β phase. The lamellar $\alpha_2(Ti_3Al) + \gamma(TiAl)$ microstructure was identified within the dendrites and the γ phase was observed in the interdendritic region, as marked in **Fig. 3b**. The lamellar $\alpha_2 + \gamma$ microstructure results from transformation of the primary β phase to the α (Ti-based solid solution with hexagonal crystal structure) phase and precipitation of the γ lamellae within the α phase, which transforms to ordered α_2 phase at lower temperatures [11]. Typical cubic symmetry of the dendrites was observed in as-cast alloy. This cubic symmetry of the dendrites is the typical feature of TiAl-based alloys solidifying through β primary phase [11, 12]. White network of the remaining β phase in the microstructure also supports the solidification of the studied alloys through the β phase. **Table 1** shows EDX chemical analysis of detected phases in the alloy Ti-47Al-8Ta.

Table 1 Chemical analysis of phases present in Ti-47Al-8Ta alloy

Ti-47Al-8Ta	Ti (at.%)	Ta (at.%)	Al (at.%)
β phase	42.65	14.63	42.72
α_2 + γ phase	42.26	9.28	48.46
γ phase	39.80	2.51	57.69

Table 2 shows measured contents of oxygen and carbon in the alloy Ti-47Al-8Ta. Generally, the alloys show a relatively low contamination by oxygen. This alloy contains up to 334 wt. ppm of oxygen. It can be seen that Ti-Al based alloy with satisfactory content of oxygen can be successfully prepared by melting in graphite crucibles and centrifugal casted into graphite moulds. The carbon content in the as-cast Ti-47Al-8Ta alloy was relatively low when compared to [13, 14], here 463 wt. ppm which corresponds to about 0.2 at.%. This amount of carbon is usually used in alloy design to improve high-temperature mechanical properties of TiAl-based alloys through solid solution and precipitation strengthening.

Table 2 Oxygen and carbon content in Ti-47Al-8Ta alloy [3]

Contents of elements	(wt. ppm)
Oxygen	334
Carbon	463

3.2. Ti-55.85Ni alloy

Melting in case of the alloy Ti-50.85Ni was performed in slightly different manner. Before preparation of the alloys from the Ti-Ni system it is sometimes recommended [2] to make the so called „flushing melting“ with use of the binary alloy Ti-50Ni, in order to create a TiC layer. This layer acts at the next melting process as a diffusion barrier, and this leads to smaller undesirable contamination of the prepared alloy with graphite from the used crucible. For melting of this alloy was used the same crucible as for preparation of the Ti-47Al-8Ta, however, material had different lumpiness and it was arranged in the crucible in a different manner (in conformity with [2]). **Fig. 4a** shows microstructure of the given alloy in the as-cast alloy, the micrograph was taken with use of an optical microscope. It is possible to see here columnar crystals, which are related to heat removal from the ingot during its cooling in the mould after casting. Optical microscopy (here in after OM) usually does not make it possible to see minuscule oxidic phases of the type Ti_4Ni_2O. For more detailed investigation of microstructural characteristics of investigated materials it was then necessary to use SEM and TEM techniques. **Fig. 4b** presents a SEM micrograph of the Ti-50.85Ni alloy. It is formed by dominant TiNi matrix, in which particles of the Ti_2Ni phase are visible. It is more difficult to observe carbidic particles with use of this method as their size is on average of approx. 0.2 - 2 µm. The alloys do not contain particles of the $TiNi_3$ phase. **Table 3** shows EDX chemical analysis of detected phases in the alloy Ti-50.85Ni.

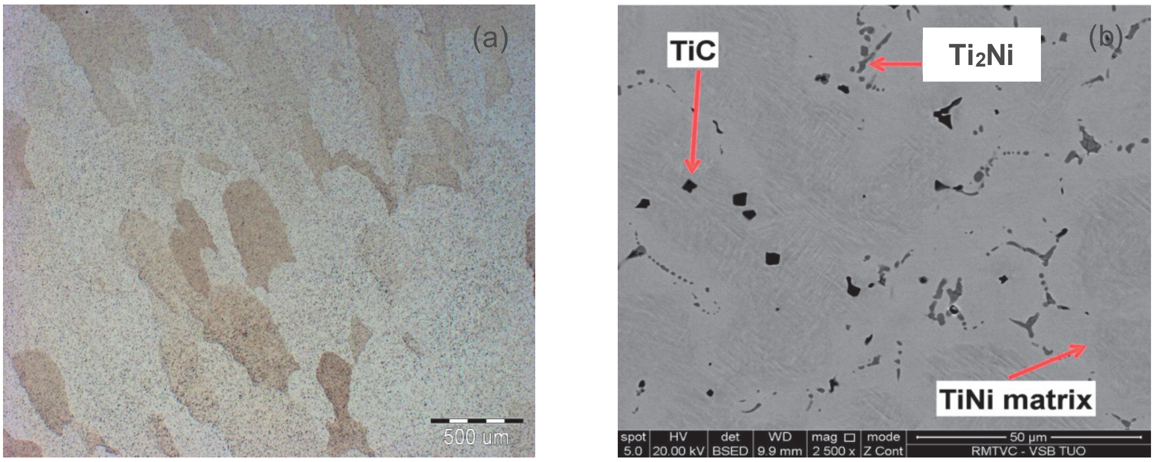

Fig. 4 OM and SEM micrographs showing microstructure of as-cast Ti-50.85Ni alloy: (a) OM, (b) SEM, Ti_2Ni and TiC carbide phases in TiNi matrix.

Table 3 Chemical analysis of phases present in Ti-50.85Ni alloy

Ti-50.85Ni	Ti (at.%)	Ni (at.%)	C (at.%)
TiNi matrix	49.35	50.65	-
Ti_2Ni	63.95	36.05	-
TiC	42.80	0.30	56.90

Table 4 shows measured contents of oxygen and carbon in the alloy Ti-50.85Ni. In this case the contents of oxygen were very similar to alloy Ti-47Al-8Ta. It can be seen that shape memory Ti-Ni based alloy with satisfactory content of oxygen can be successfully prepared by melting in graphite crucibles and centrifugal casting into graphite moulds. The carbon content in the as-cast Ti-50.85Ni alloy is relatively higher when compared to [2].

Table 4 Oxygen and carbon content in Ti-50.85Ni alloy

Contents of elements	(wt. ppm)
Oxygen	384
Carbon	613

Both alloys were after metallurgical process subjected to the radiographic examination by Vítkovice Testing Center, lab. No. 1036. Parameters of the examination were following: Film size / type: 10x30 cm / T200, source - surface distance 50 cm, material thickness: diameter 20 mm, exposition time / dose: 2.78 Ci.hod, sensitivity: W13, image quantity indicator: 10, source: ^{192}Ir. By this testing method was found, that ingot contains no gas porosity, sand and slag, shrinkage, cracks, hot tears and inserts.

4. CONCLUSION

Two different types of experimental alloys were prepared in vacuum induction furnace in graphite crucible. The melting conditions and the crucibles were almost the same. Casting of material was performed by centrifugal casting into a non-preheated graphite mould. Prepared ingots were subjected to the radiographic examination. It was found, that no large casting defects were present in the material - castings were absolutely satisfactory. The Ti-Ni based alloy can be hot swaged. By determination of gas contents (oxygen) it was found, that the alloy Ti-47Al-8Ta was slightly cleaner when compared with the alloy Ti-50.85Ni. In case of carbon content determination, better results were obtained during preparation of Ti-47Al-8Ta. From the perspective it was found that microstructures of both alloys were in accordance with the current knowledge. It should be noted that this type of melting and casting of selected alloys can be characterized as suitable.

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