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Structure and properties of arc melted dilute nial intermetallic compounds

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ABSTRACT

In this study, the effect of nonsoluble alloying elements on the structure and properties of nickel aluminide (NiAl) was investigated. NiAl and dilute NiAl-X (X=Ta, Nb, Mo) alloys with 1.0 and 2.0 at. % solute contents were arc melted under argon atmosphere using water cooled copper mold. High temperature oxidation test were done at 1000°C for 5, 15, 25 and 35h. Scanning electron microscope, X-ray diffractometer and energy dispersive X-ray spectroscopy were used to characterize the alloys. Alloying elements increased the density and hardness of NiAl. From the structural analyses, it was determined that $TaNi_2$, Nb and Mo phases formed in the cast alloys. Additionally, alloying elements developed texture such that Ta and Nb promoted [110] orientation while Mo promoted [100] orientation. Fracture analyses of the samples showed that cleavage type fracture occurred in all of the alloys. Tantalum containing alloys were found to have better high temperature oxidation resistance than the other alloys.

1. Introduction

NiAl intermetallic compound is preferred for high temperature applications in automotive, aerospace, power generation industries, etc. due to its low density, high melting temperature, high thermal conductivity, high oxidation and corrosion resistance [1-4]. However, low ductility and fracture toughness at low temperatures as well as low strength and creep resistance at high temperatures limit its technological applications significantly [5]. Therefore, several attempts have been made to optimize the mechanical properties of NiAl. Many of these studies were focused on the microstructural control by alloying elements and processing techniques including mechanical alloying [6-8], rapid solidification [9-11] and directional solidification [12-14].

The high temperature strength and creep resistance of NiAl were mainly improved with solid solution strengthening and precipitation of Heusler/Laves phases. For this purpose, many researchers [15-18] investigated the effect of refractory elements, including Hf, Ti, Nb, Ta, Mo and Cr etc. in polycrystalline NiAl intermetallic compound. Several researchers [19-24] introduced refractory and rare earth elements (Ho, Dy, etc) to NiAl eutectic alloys to improve creep strength further with solid solution and precipitation hardening mechanisms. Further improvements in mechanical properties of NiAl were obtained with grain refinement using rapid solidification technique.

In this study, Ta and Nb alloying elements were chosen due to their positive contribution in creep properties of NiAl, while Mo alloying element was chosen due to its ability to improve room temperature ductility of NiAl. After processing dilute NiAl-X compounds, the microstructure, density, hardness, high temperature oxidation resistance and fracture behavior of rapidly solidified NiAl and dilute NiAl-Ta, NiAl-Nb and NiAl-Mo intermetallic compounds were investigated.

2. Material And Method

2.1. Materials and sample preparation

Intermetallic compounds were prepared from metal powders. Metal powders with 99.8-99.98 % purities were obtained from Alfa Aesar. After weighting the powders in defined ratios and mixing them physically, they were cold pressed to obtain compact samples with 14 mm in diameter and 12 mm in thickness. Afterwards, the samples were arc melted in a water cooled copper mold under high purity argon atmosphere. During melting, arc current was 200A. In order to obtain homogenous compositions, each sample was melted three times. The composition of the intermetallic compounds is given in Table 1.

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Table 1. Composition of the intermetallic compounds.

Compound	Composition (at %)				
	Ni	Al	Ta	Nb	Mo
IMET-1	50	50	-	-	-
IMET-2	49.5	49.5	1	-	-
IMET-3	48	48	2	-	-
IMET-4	49.5	49.5	-	1	-
IMET-5	48	48	-	2	-
IMET-6	49.5	49.5	-	-	1
IMET-7	48	48	-	-	2

2.2. Metallographic sample preparation

Each sample was cut half and the cross sections were prepared using 240, 320, 400, 600 and 800 grit SiC papers. After grinding, each surface was polished using 6µm and 3µm diamond solutions. Finally, to reveal the microstructural details, each sample was etched in 5% HNO_3-water solution for 5-10 min.

2.3. Density measurements

The density of the samples was determined according to the Archimedes' principle by using Presica XB 220-A balance with 1/10000 precision. For each sample, three measurements were done and then an average density was determined.

2.4. Hardness measurements

Rockwell A hardness (HRA) of the samples was measured using Affri System VRSD–251 model universal macro hardness device. For each sample, four measurements were done and their average was taken as hardness.

2.5. Microscopic and chemical analyses

Surface morphology and fracture surface of the samples were characterized using JEOL JSM6060 and FEI QUANTA 400 FEG scanning electron microscopes (SEM), respectively. Chemical compositions of the phases were determined using energy dispersive X-ray spectroscopy (EDX). Due to small sample dimensions, impact tests were simulated as follows; half of the samples were placed in a clamp and the upper part of the unnotched samples was broken with a single hammer hit as it was in izod impact test.

2.6. Structural analysis

X-ray diffractometer (XRD) was used to determine the present phases in the samples. XRD measurements were performed at a 4^o/min scan rate by using a Rigaku D-Max RINT-2200 series X-ray diffractometer operated at 40 keV and 40 mA.

2.7. Oxidation tests

High temperature oxidation resistance of the intermetallic compounds was determined by heat treating the samples in normal atmosphere for 5 h, 15 h, 25 h and 35 h at 1000^oC and then determining weight gains.

3. Results And Discussion

The measured density of NiAl is 5.53 g/cm^3 which is close to the theoretical density of 5.65 g/cm^3. Alloying element and solute content increased the density and hardness of the alloys. The density and hardness of the intermetallic compounds are given in Table 2. As it is seen from the table, percent increase in the density of NiAl is between 2.17 and 14.29. Similarly, increase in the hardness of NiAl is between 9.92% and 14.33%. Among the alloying elements, Ta addition has the largest effect and Mo has the lowest effect on the density and hardness of NiAl. The fact that Mo containing alloys have lower hardness than the others implies that the ductility of NiAl-Mo alloys is slightly higher than NiAl-Ta and NiAl-Nb alloys. Since the density of the alloys is important in terms of energy consumption, specific hardness of the alloys was determined by taking hardness to density ratio. It was determined that dilute NiAl alloys with low solute content had higher specific hardness than the alloys with high solute content. Among the alloys, dilute NiAl-Nb alloys have the best combination of hardness and density. Since mechanical strength increases with hardness, it can also be said that dilute NiAl-Nb alloys have better combination of strength and density.

Table 2. Density and hardness of the intermetallic compounds.

Compound	Density (g/cm^3)	% Increase	Hardness (HRA)	% Increase	Specific Hardness
IMET-1	5.53	-	63.5	-	11.48
IMET-2	6.14	11.03	71.4	12.44	11.63
IMET-3	6.32	14.29	72.6	14.33	11.49
IMET-4	5.65	2.17	70.2	10.55	12.42
IMET-5	5.88	6.33	71.7	12.91	12.19
IMET-6	6.05	9.40	69.8	9.92	11.54
IMET-7	6.16	11.39	70.9	11.65	11.51

The microstructure of the alloys with low solute content and EDX spectrums taken from the grain boundary phases are given in Figure 1. As it is seen from the micro images, dilute NiAl intermetallic compounds have polycrystalline structure and metallic phases precipitated at the grain boundaries. Both NiAl-1.0 at.% Ta and NiAl-1.0 at.% Nb have very similar grain structure. However, NiAl-1.0 at.% Nb alloy has larger mean grain size than NiAl-1.0 at.% Ta. On the other hand, NiAl-1.0 at.% Mo has slightly different grain structure compared to the others in terms of grain shape and grain boundary phases. The black dots on the images indicate surface contamination occurred during sample preparation. From the EDX spectrums it was determined that Ta, Nb and Mo elements were rich in grain boundary phases. Since similar microstructures were observed from the intermetallic compounds with high solute content, they were not reported here.

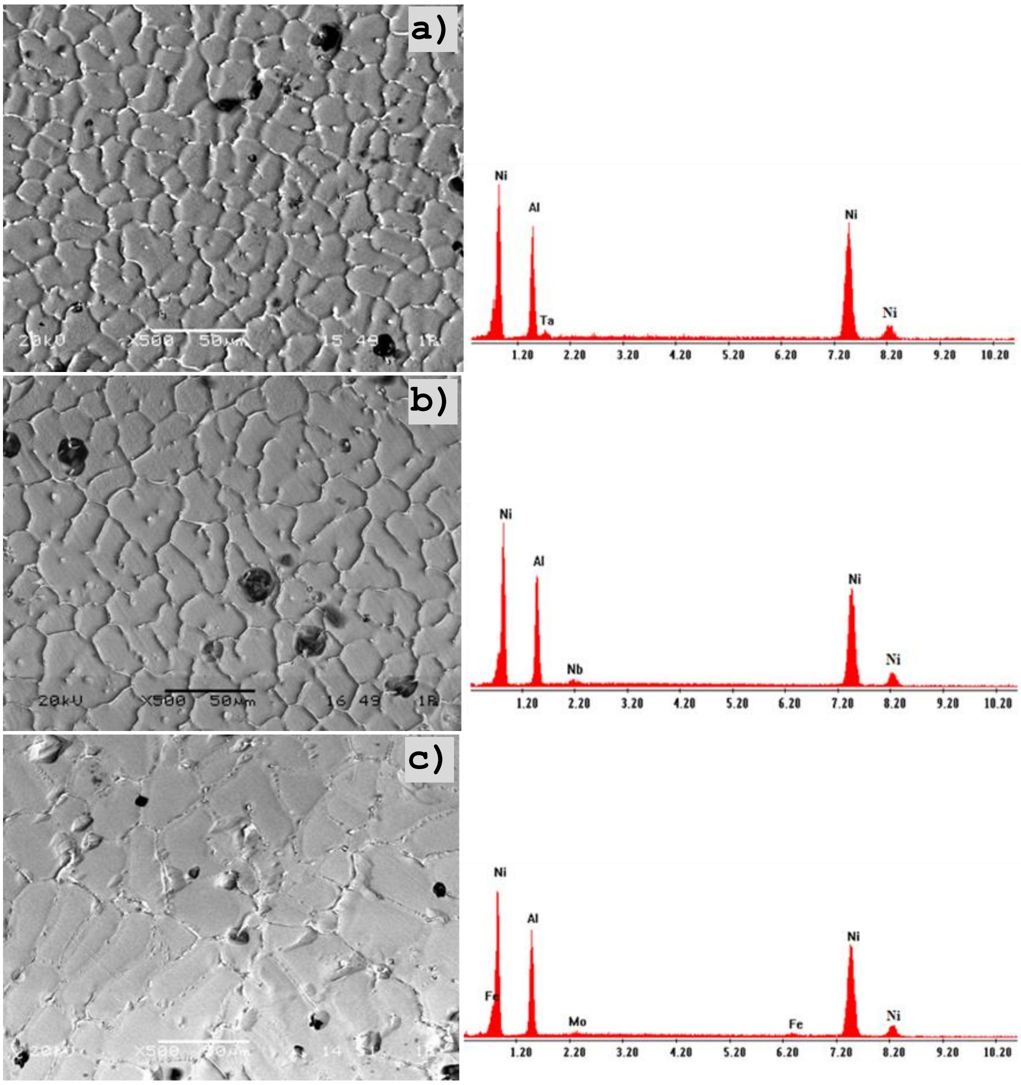

Figure 1. Microstructure of the dilute NiAl alloys and EDX spectrums taken from grain boundaries; a) NiAl-1 at% Ta, b) NiAl-1 at% Nb and c) NiAl-1 at% Mo.

X-ray diffraction patterns of NiAl and dilute NiAl-X compounds with low solute content are given in Figure 2. In order to show the XRD patterns of the samples together, the graphs were shifted. From X-ray analyses it was determined that Ni_2Ta and Mo phases formed in Ta and Mo containing samples respectively. As it can be seen from the XRD patterns, alloying elements develop texture since (111) and (211) peaks are absent in the XRD graphs of dilute NiAl compounds. Therefore, it can be said that Ta and Nb elements promote [110] orientation because (110) peak is much stronger than the other peaks. Similarly, Mo promotes [100] orientation. On the other hand, NiAl crystals have random crystal orientations since all diffractions are present in the pattern. Since similar XRD patterns were observed from the intermetallic compounds with higher solute content, they were not reported here.

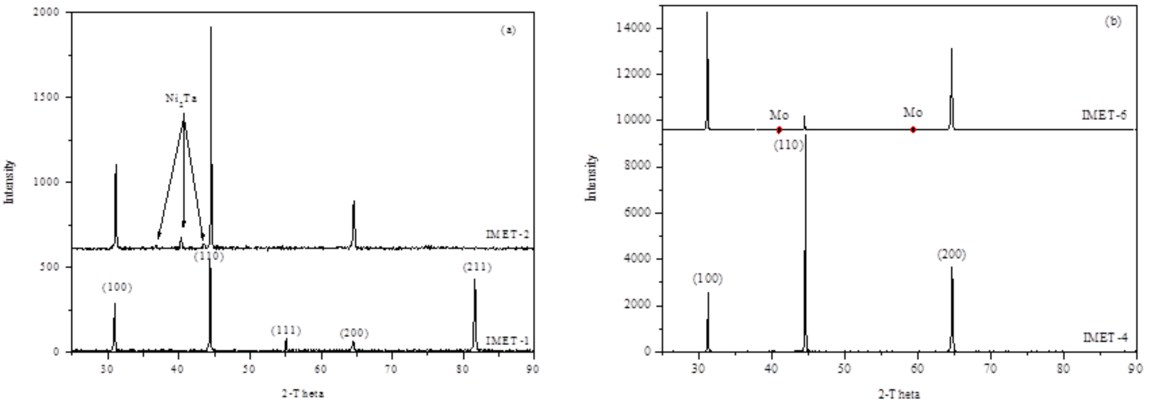

Figure 2. XRD patterns of NiAl and dilute NiAl alloys; a) IMET1-IMET2 and b) IMET4-IMET6.

The results obtained here are consistent with the earlier studies done by Pathare [25], Xiaojun et al. [26] and Liu et al. [27]. Pathare studied the effect of Ta and Nb alloying elements at 1 and 2 at. % on the structure and properties of extruded NiAl. In this study, it was shown that both 1 at. % Ta and Nb containing NiAl alloys had single phase structure, while 2 at. % Ta and Nb containing alloys had non-uniform distribution of elongated Ta or Nb rich second phases at the grain boundaries. This is the structure we observed in Ta and Nb containing samples. Xiaojun et al. studied the microstructural evolution of NiAl-2.5Ta-7.5Cr alloy during annealing at different temperatures. They observed coarse Laves phase $Ta(CrNiAl)_2$ in as-cast alloy which is similar to $TaNi_2$ Laves phase that we observed in NiAl-2Ta system. Liu et al. investigated the effect of several alloying elements including Mo on the structure and properties of NiAl. It was shown that Mo addition improved room temperature ductility and high temperature strength.

The fracture surfaces of NiAl and dilute NiAl compounds with high solute content are given in Figure 3. The microscopic appearance of the fracture surface of NiAl is given in Figure 3a. The cleavage facets and steps in the facets in Figure 3a indicate that cleavage type fracture occurred in NiAl. It is known that cleavage type fracture occurs along low-index crystallographic planes. Depending on the loading such as tension or impact, significant or little plastic deformation may occur during cleavage fracture. Similar facet and steps were also observed in dilute NiAl alloys.

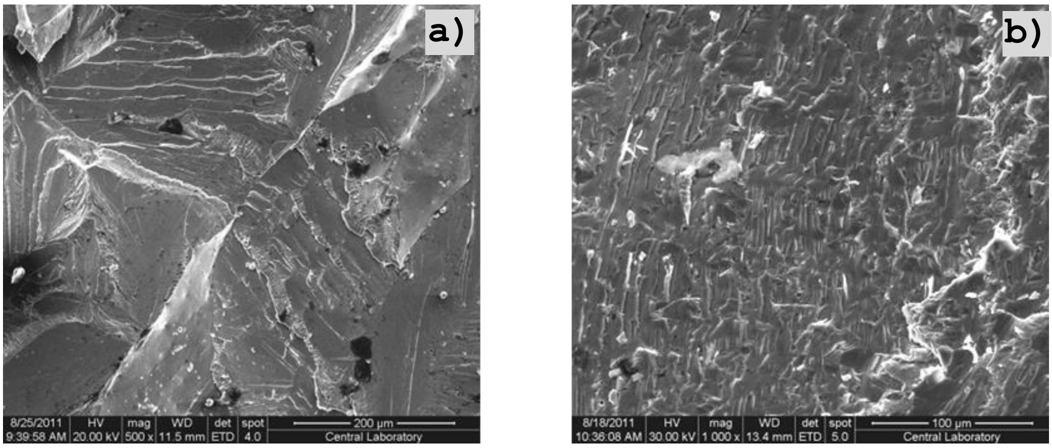

Figure 3. Fracture surfaces of the intermetallic compounds; a) NiAl, b) NiAl-2.0 at% Ta, c) NiAl-2.0 at% Nb and d) NiAl-2.0 at% Mo.

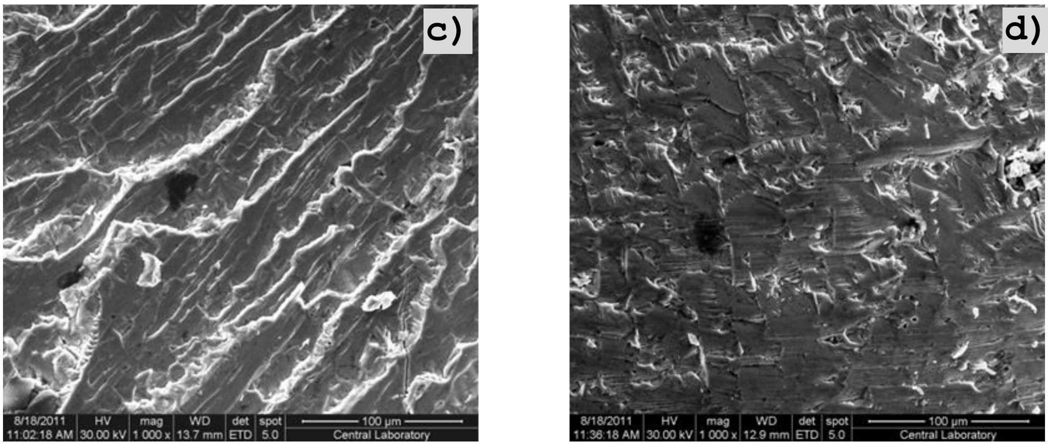

Figure 3. (Continued) Fracture surfaces of the intermetallic compounds; a) NiAl, b) NiAl-2.0 at% Ta, c) NiAl-2.0 at% Nb and d) NiAl-2.0 at% Mo.

High temperature oxidation resistance of the alloys was studied at 1000°C for 5 h, 15 h, 25 h and 35 h. Initial weight, final weight and weight gain of the samples are given in Table 3 and oxidation rate of the intermetallic compounds is given in Figure 4. As it can be seen from the figure, Ta containing alloys have better high temperature oxidation resistance than other alloys. C.T. Liu et al. [27] showed that 0.4 at. % Ta addition to NiAl 1,5Mo at 1000°C significantly increased the oxidation resistance of the alloy.

Table 3. Weight of the intermetallic compounds before and after oxidation tests at 1000 °C.

Compound	Initial Weight (g)	Final Weight (g)				Weight Gain (g)			
		5 h	15 h	25 h	35 h	5 h	15 h	25 h	35 h
IMET-1	2.236	2.236	2.242	2.292	2.307	0	0.006	0.056	0.071
IMET-2	2.333	2.335	2.341	2.354	2.354	0.002	0.008	0.021	0.021
IMET-3	1.818	1.822	1.823	1.825	1.824	0.004	0.005	0.007	0.006
IMET-4	2.222	2.233	2.259	2.329	2.358	0.011	0.037	0.107	0.136
IMET-5	1.987	1.992	2.029	2.157	2.188	0.005	0.042	0.170	0.201
IMET-6	1.453	1.468	1.512	1.609	1.635	0.015	0.059	0.156	0.182
IMET-7	1.194	1.210	1.230	1.264	1.271	0.016	0.036	0.070	0.077

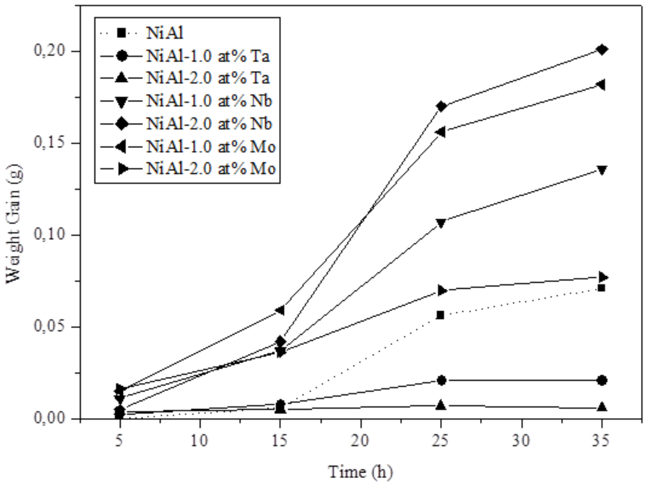

Figure 4. High temperature oxidation of NiAl and dilute NiAl alloys.

4. Conclusions

In this study, the effect of nonsoluble alloying elements on the structure and properties of NiAl was investigated. NiAl and dilute NiAl alloys were prepared using arc melting and water cooled copper mold. Alloying NiAl resulted in higher density and hardness. When specific hardness of the alloys was compared, it was found that dilute NiAl-Nb alloys had better combination of density and strength.

Microstructural analyses showed that dilute NiAl alloys had polycrystalline structure and second phases precipitated at the grain boundaries. Alloying elements developed texture such that Ta and Nb promoted [110] orientation while Mo promoted [100] orientation. From the fracture surfaces of NiAl and dilute NiAl alloys it was seen that cleavage type fracture occurred in all of the alloys. Among the alloys, Ta containing alloys had the best high temperature oxidation resistance.

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