

EFFECT OF ALLOYING ELEMENTS ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF NiAl-Cr(Mo) EUTECTIC ALLOY

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Abstract: Microstructure and mechanical properties of NiAl-Cr(Mo) eutectic alloy and the effect of 0.1Fe, 1Nb, 4Ti additions are investigated at room temperature (RT) and high temperature (1000°C). The alloys are prepared by arc melting method and then homogenized at 1300°C in Ar atmosphere for 24 h. In addition to the room temperature and high temperature compression tests and X-ray diffraction measurements, hardness measurements and microscopic analyses at room temperature are performed. NiAl-Cr(Mo) alloy exhibit fine cellular eutectic structure with coarse eutectic at interdendritic regions. However, the Nb and Ti addition reduced the formation of eutectic structure. Cr₂Nb-type Laves and Ni₂AlTi-type Heusler phases are observed in the XRD patterns of the Nb and Ti containing alloys respectively. Addition of Fe does not have important effect on the room and high temperature yield strength of the alloy, while the Nb and Ti increase the yield strength of the alloys at both temperatures.

Keywords: NiAl-Cr(Mo), LAVES PHASE, HEUSLER PHASE, MECHANICAL PROPERTIES

1. Introduction

Ordered intermetallic materials have taken considerable attention for high temperature structural applications last two decades. Among the ordered intermetallics, NiAl intermetallic compound with B2 crystal structure has excellent properties including high melting point, low density, good thermal conductivity and excellent oxidation resistance up to 1300°C [1–5]. Such attractive properties make NiAl an attractive candidate for high temperature structural applications. However, low ductility and fracture toughness at room temperature and poor elevated temperature strength of the NiAl limit its commercial applications significantly [6,7]. Some strengthening and toughening approaches such as alloying, second-phase strengthening and single crystal preparation were proposed to overcome the drawbacks of NiAl [8–12]. Previous studies on NiAl revealed that the additions of refractory metals like Cr, Mo, V and Ta, etc. can improve the room temperature toughness and elevated temperature strength of NiAl simultaneously [13–15]. Walter et al. [13] showed that directionally solidified NiAl–Cr eutectic alloy strengthened by the Cr fiber had better toughness and creep strength than NiAl. In addition, Cline et al. [16,17] showed that adding Mo to the NiAl–Cr eutectic alloy would lead to a change of microstructure from Cr fibers to Cr(Mo) lamellae.

Among the eutectic alloys, NiAl–28Cr–6Mo alloy is very attractive due to its good room temperature ductility and fracture toughness as well as good high temperature strength [18]. The low temperature and high temperature mechanical properties of NiAl–28Cr–6Mo eutectic alloy can be further improved with micro alloying. Therefore, it is of interest to investigate the room and high temperature microstructure and mechanical properties of Fe, Nb and Ti containing NiAl–Cr(Mo) eutectic alloys since there are not enough studies on the arc melted and micro alloyed eutectic alloys. Here, we present low and high temperature microstructure and mechanical properties of arc melted NiAl–28Cr–6Mo, 65.99(NiAl)-28Cr-6Mo-0.1Fe and Ni(33-X)Al-28Cr-6Mo-X (X=1 at % Nb, 4 at % Ti) eutectic alloys.

2. Experimental Procedures

NiAl–28Cr–6Mo eutectic alloy with X=0.1 at % Fe, 1 at % Nb and 4 at % Ti nominal composition are prepared by vacuum arc melting using Edmund Buhler Vacuum Arc Melter. The purity of metal powders used in this work is 99.9 % for Ni, 99.9 % for Al, 99.99 % for Cr, 99.95 % for Mo, 99.98 % for Fe, 99.99 % for Nb and 99.9 % for Ti. Before melting the alloys, the mixture of metal powders are cold pressed. All arc melted buttons are turned over and remelted at least four times to get homogeneous specimens. In addition, homogenization heat treatment of the alloys has been performed at 1300°C for 24 hours in Ar atmosphere (40 L/h). After that, all alloys are furnace cooled. Before the homogenization heat

treatment, the atmosphere of the furnace is purged three times by filling high purity Ar and then vacuuming to 6.5×10^{-2} mbar. Since the weight loss for each sample is less than 0.5%, the alloy compositions have been considered to be equal to their nominal compositions.

Wire electro-discharged machine (EDM) is used to get specimens for microstructure analyses and compression tests. For microstructure analyses, specimens are cut along the longitudinal and transverse directions of arc melted buttons. The sections are mechanically polished and then etched in a 5 % nital solution. The microstructures are observed by optical microscope (OM) and scanning electron microscope (SEM, Carl Zeiss Ultra Plus Gemini FESEM) with attached energy-dispersive X-ray spectrometer (EDS). The room temperature and high temperature (1000°C) X-ray diffraction (XRD, Rigaku Ultra IV diffractometer) measurements have been conducted at a rate of 4°/min. between 25°–85° using 40kV and 30mA. The Brinell hardness (HB2.5/31.25/10) of the alloys is determined using Qness/Q250M hardness device. For each alloy, the average hardness of the alloys is obtained from three hardness measurements. In compression tests, cylindrical specimens with 3 mm in diameter and 5 mm in height have been used. The room temperature compression tests are performed at a strain rate of $5 \times 10^{-4} \text{ s}^{-1}$ in a Zwick/Roell Z600 static/dynamic test machine. The high temperature (1000°C) compression tests are performed at a strain rate of $5 \times 10^{-3} \text{ s}^{-1}$ in a Shimadzu AG-IS 250 test machine.

3. Results and Discussion

3.1. Microstructure characteristics

Fig. 1 shows the typical SEM microstructures of four alloys produced for this study. In the first and second alloy (Fig.1 a and b) consisted of NiAl and Cr(Mo) phases and the structure was fully eutectic. In the eutectic cells, dark NiAl and gray Cr(Mo) lamellas radiate from the cell center to the cell boundaries. The interlamellar spacing of the Cr(Mo) and NiAl phases near the cell center is smaller than that near the cell boundary. Some coarse and irregular plates formed at the cell boundaries. Figure 1. c and d show the microstructure of the Nb and Ti bearing alloys respectively. In addition to the eutectic phases, a Nb-rich phase formed in the Nb bearing alloy. Introduction of Ti reduced the formation of lamellar microstructure. Ti addition essentially changed the structure of NiAl-Cr(Mo) alloy by forming Cr(Mo) rich dendrites and Ti bearing NiAl solid solution.

The EDS point analyses taken from all alloys are given in Fig. 2. NiAl-Cr(Mo) eutectic and Fe added alloy show similar results except that small amount of Fe distribution in eutectic phases. Existence of Ni and Al in the Cr(Mo) phase and Cr and Mo in the NiAl phase show that small NiAl precipitates are present in the Cr(Mo) phase and similarly Cr(Mo) precipitates exist in the NiAl

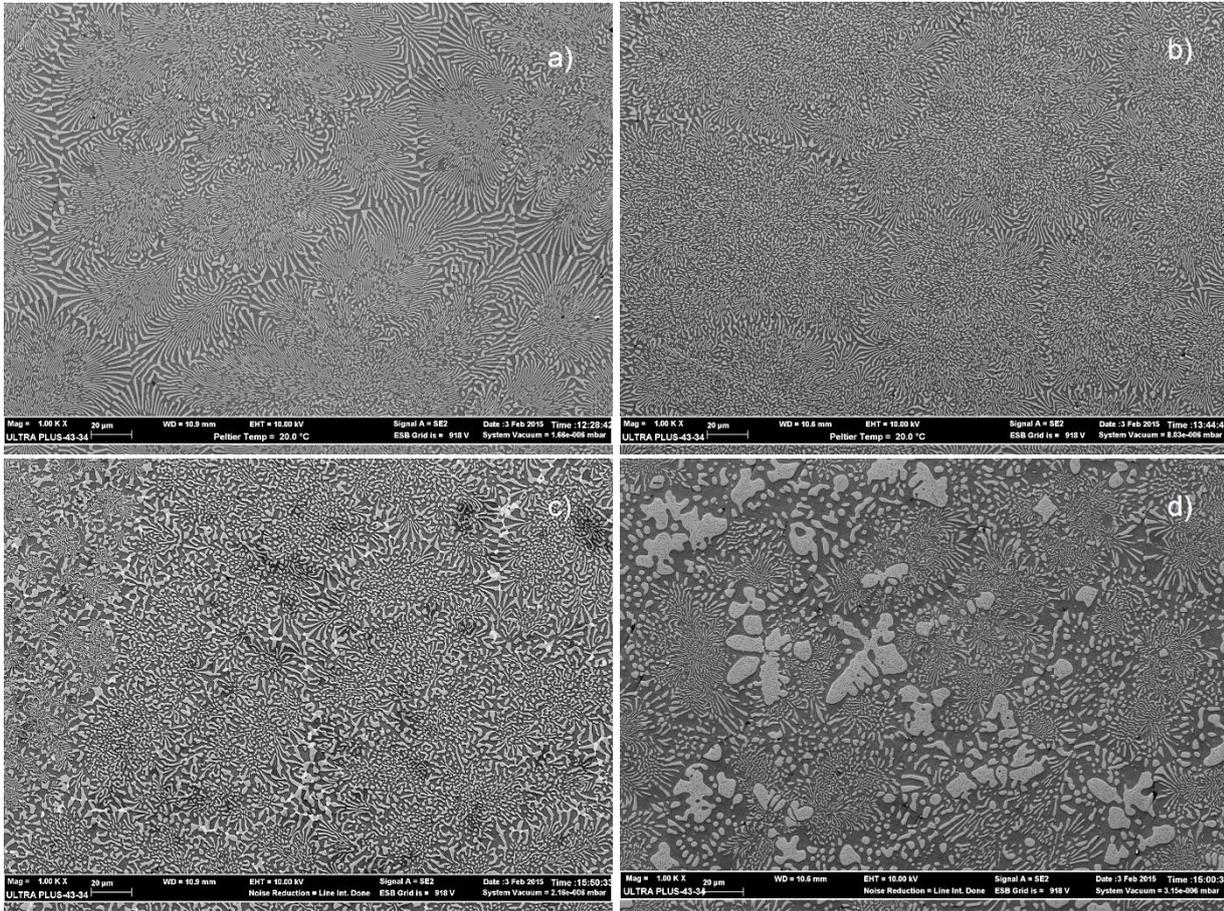
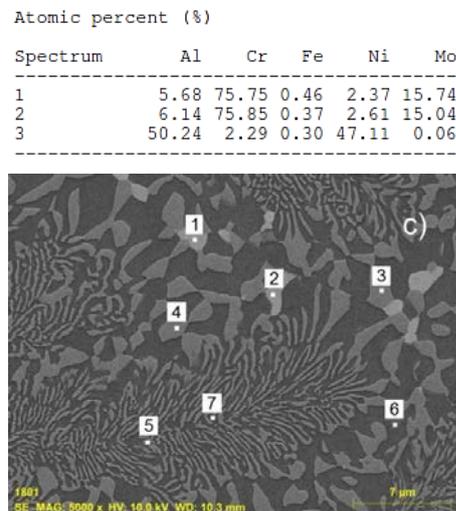
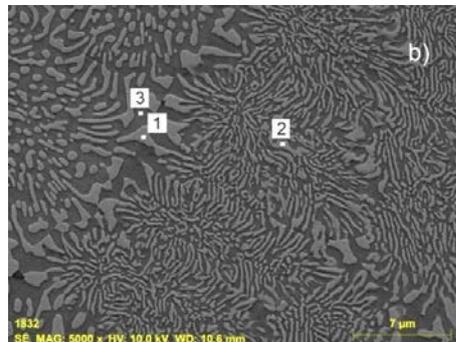
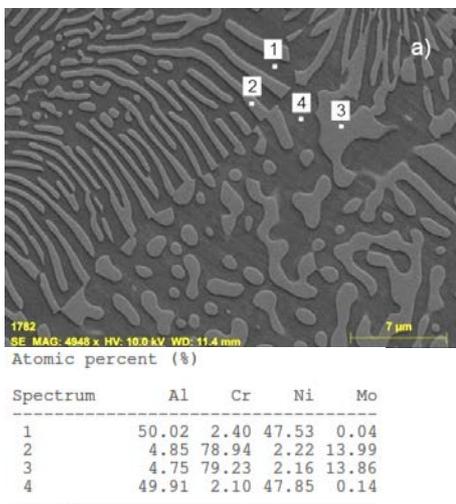


Fig. 1 The microstructure of alloys; a) NiAl-Cr(Mo) b) NiAl-Cr(Mo)-0.1Fe c) NiAl-Cr(Mo)-1Nb and d) NiAl-Cr(Mo)-4Ti.

phase [19,20]. The fact that chemical composition of the Cr(Mo) and NiAl phases in near eutectic cell center and interdendritic region are very similar indicates that the alloys are homogeneous. As it is seen from the point analyses, Fe partitions both Cr(Mo) and NiAl phases. However, Fe participates into the Cr(Mo) phase slightly more than the NiAl phase as it is given from the Fig.1b. It can be seen from Fig. 2c, the microstructure is composed of NiAl, Cr(Mo) and Cr₂Nb phases. The Cr₂Nb Laves phase formation in the Nb content alloy breaks down the cellular eutectic structure formation. The Fig.2d EDX spectra revealed that dark phase (matrix) enriched in Ni, Al, Ti and grey phase in Cr, Mo. However, both phases have small quantities of other elements.



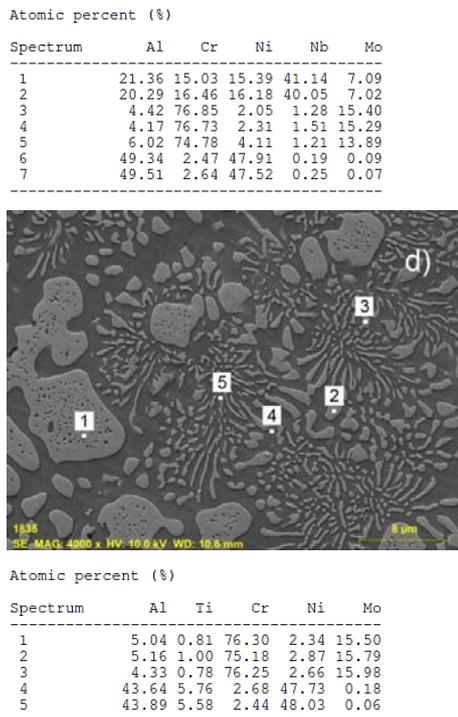


Fig. 2 The EDX points of alloys; a) NiAl-Cr(Mo), b) NiAl-Cr(Mo)-0.1Fe, c) NiAl-Cr(Mo)-1Nb and d) NiAl-Cr(Mo)-4Ti.

Phase analyses of the alloys were performed at room temperature and 1000°C. The XRD patterns of the alloys are given in Fig. 3. The room temperature x-ray patterns of the alloys show that the all alloys have NiAl and Cr(Mo) phase structure. These phases were also observed by other researchers in NiAl-Cr(Mo) eutectic alloys containing Ti, Hf, Nb alloying elements [20–22]. In addition to these phases, the Nb containing alloy has Cr₂Nb, Laves phase and Ti containing alloy has Ni₂AlTi, Heusler phase. At 1000°C, the peaks slightly shift to lower Bragg angles indicating that lattice parameters of the phases increase. Increase in lattice parameters may be a result of increase in the separation of planes at high temperatures. In addition, at high temperature XRD patterns of NiAl-Cr(Mo) and 4Ti containing alloys, a peak does not belong to NiAl and Cr(Mo) phases is observed at 2-Theta 33.7°. Since high temperature x-ray data base is not available, phase diagrams were used to interpret the anomalous peak. First of all, the peak is not due to Ti addition because the peak is also present in NiAl-Cr(Mo). Based on the phases in Ni-Al-Cr-Mo systems at 1000°C, it is possible that beta-NiMo is formed at 1000°C.

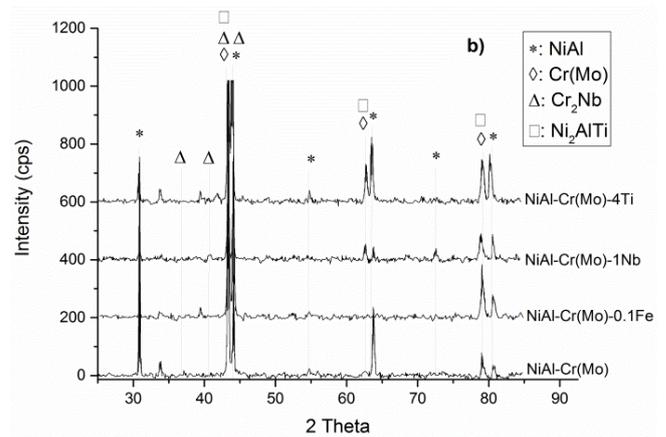
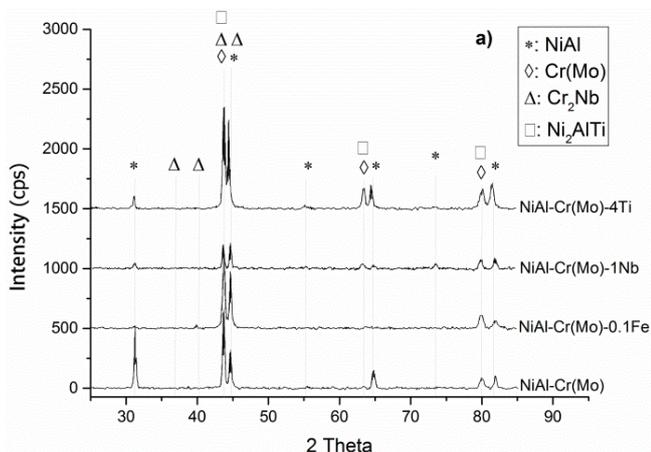


Fig. 3 XRD patterns of the alloys a) at room temperature and b) at high temperature.

3.2. Hardness properties

Macro hardness test was used instead of micro hardness since penetration depth and area are small due to small applied loads in micro hardness tests. Therefore, it is not possible to obtain a representative hardness value of the alloys with micro hardness measurements. As a result, Brinell hardness of the homogenized alloys was measured by applying 31.5 kg load for 10 s on 2.5 mm steel ball (HB2.5/31.25/10). The hardness values of the alloys are given in Table 1.

As it is seen from the Table 1, Ti added alloy has the highest hardness. Nb containing alloy has similar hardness with the NiAl-Cr(Mo) base alloy but the hardness of Fe containing alloy is lower than that. Based on the hardness of the alloys, the average diameter of the indentations is 380 µm for NiAl-Cr(Mo).

Table 1: Brinell Hardness of the eutectic alloys.

Alloy	HB Measurements			Average HB
NiAl-Cr(Mo)	283	270	269	274
NiAl-Cr(Mo)-0.1Fe	259	258	255	257
NiAl-Cr(Mo)-1Nb	286	276	266	276
NiAl-Cr(Mo)-4Ti	402	398	402	401

The Brinell hardness measurement also provided a valuable information on the deformation behaviour of the phases, ductility and toughness of the alloys. After Brinell hardness measurements, the surface of the alloys was analysed using SEM in order to determine presence of surface cracks. As it can be seen from the SEM images given in Fig. 4, plastic deformation occurs during indentation and any big surface cracks are not observed. Therefore, it can be said that all alloy have enough ductility and toughness. However, in a closer look, micro cracks formed during indentation in 0.1 Fe containing eutectic alloy. From microscopic analyses, it can be said that the Cr(Mo) phase behaves as barrier to crack propagation. As a result, the Cr(Mo) phase contributes to the fracture strength of the alloys by absorbing crack energy and slowing down the crack growth rate. Crack formation in the alloys can be explained with the ductility and toughness of the alloys. When a force is applied on the hard metallic phase, it transfers the applied load to the ductile matrix. The metal matrix absorbs the energy by going through plastic deformation. If the stress is larger than the fracture strength of the matrix, crack initiation in the matrix occurs.

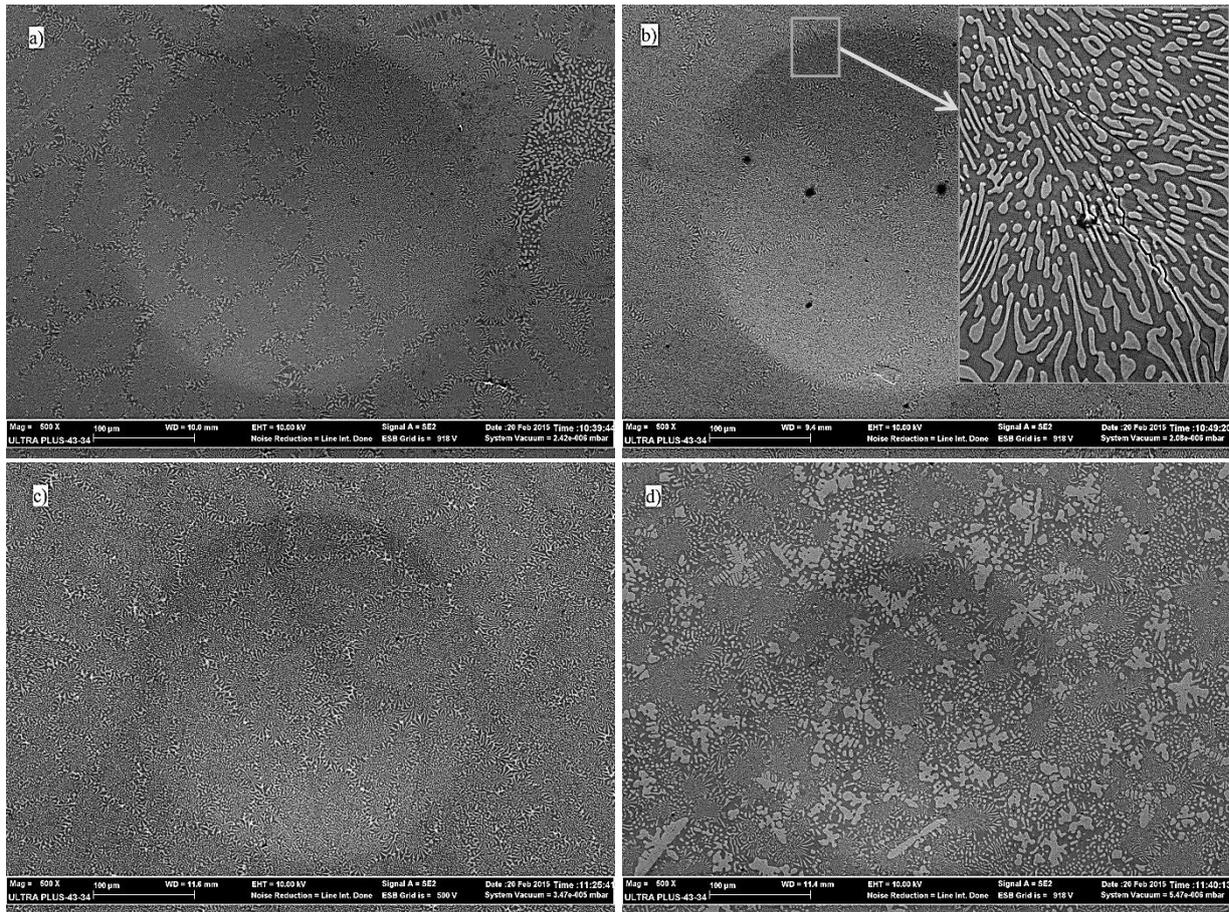


Fig. 4 Deformations on the indentation for; a) NiAl-Cr(Mo), b) NiAl-Cr(Mo)-0.1Fe, c) NiAl-Cr(Mo)-1Nb and d) NiAl-Cr(Mo)-4Ti.

3.3. Compressive properties

Mechanical properties of the alloys were determined by compression tests at room temperature and 1000°C. The stress-strain curves are given in Fig. 5 and mechanical properties of the alloys are given in Table 2.

The modulus of elasticity of the alloys given in this table is taken from the slope of the stress-strain curves. As it is seen from the room temperature data, Ti bearing alloy shows very high ductility, yield strength and ultimate compression strength. The

strength of the NiAl-Cr(Mo) eutectic alloy increases with Ti addition dramatically. However, the Fe addition slightly decreases the ultimate compression strength and strain, moreover Nb added alloy shows the lowest strain. Increasing the test temperature to 1000°C results in much lower mechanical properties. Among the alloys, Nb containing alloy shows the highest yield strength and ultimate compression strength. The increase in strength can be attributed to the formation of Cr_2Nb Laves phases.

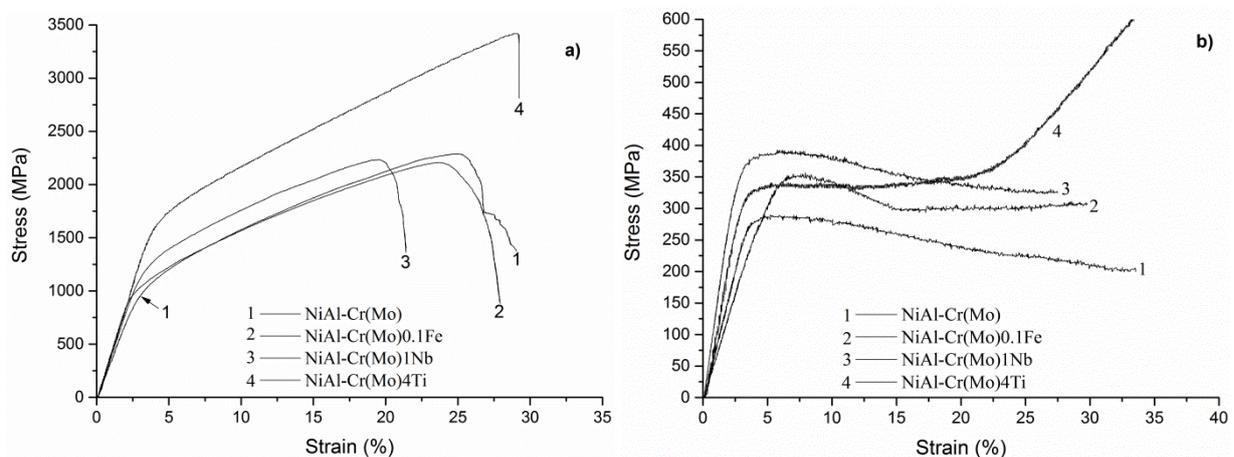


Fig. 5. Compression stress-strain curves, a) at room temperature and b) at 1000°C.

Table 2. The room temperature and high temperature compressive properties of the alloys.

Alloy		E (GPa)	σ_y (MPa)	ϵ_y (%)	σ_u (MPa)	ϵ_u (%)
NiAl-Cr(Mo)	RT	36.58	902	2.68	2285	24.33
	1000 °C	8.95	268	3.17	292	5.6
NiAl-Cr(Mo)-0.1Fe	RT	43.82	949.9	2.36	2206.1	23.38
	1000 °C	7.48	261.9	3.71	357	6.80
NiAl-Cr(Mo)-1Nb	RT	41.16	1120	2.91	2231	19.06
	1000 °C	14.96	325	2.39	398	5.9
NiAl-Cr(Mo)-4Ti	RT	46.15	1548.5	3.6	3430.3	27.71
	1000 °C	12.09	293.2	2.6	340	-

4. Conclusions

The room temperature and high temperature microstructure and mechanical properties of arc melted NiAl-Cr(Mo) eutectic and 65.99(NiAl)-28Cr-6Mo-0.1Fe, Ni(33-X)Al-28Cr-6Mo-X (X=1 at % Nb, 4 at % Ti) alloys are studied. NiAl-Cr(Mo) eutectic alloys have fully lamellar eutectic structure. The Fe addition results in coarser interdendritic regions and lower volume fraction of fine eutectic cells. The eutectic structure changed effectively with the addition of Nb and Ti. Both of them reduce the cellular eutectic structure formation. The EDX analyses reveal that Fe partitions both the NiAl and Cr(Mo) phases, Nb partition mainly to the Cr(Mo) and Ti partition to the NiAl. The room temperature and high temperature XRD measurements show that NiAl-Cr(Mo) and Fe added alloys have two-phase structure, NiAl and Cr(Mo). A third phase as Cr₂Nb and AlTi were seen at Nb and Ti added alloys respectively. In addition, high temperature XRD patterns show that peaks shifted to the left indicating that lattice parameters of the phases increase and there is a possibility of beta-NiMo formation at 1⁰⁰⁰°C.

All alloys exhibit very high yield strength and total strain at the room temperature. At high temperature, mechanical properties of the alloys decrease significantly. Addition of Fe does not have important effect on the room and high temperature yield strength of alloy while the Nb and Ti increase the yield strength of the alloys at both temperature. Among the alloys, Nb containing alloy has the highest high temperature yield strength, while Ti containing alloy has the highest room temperature yield strength.

Acknowledgments

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