

INFLUENCE OF COARSE SECOND PHASE ADDITIONS ON MECHANICAL PROPERTIES OF NiAl

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ABSTRACT

NiAl/Nb and NiAl/Cr composite materials were prepared by a powder metallurgical approach. The content of the second phases varied between 5 and 10 weight percent. The thermodynamic stability of Nb and Cr particles in the NiAl matrix was studied at 1473K (1200°C) by means of SEM, XRD and microhardness measurements. In the case of Nb the formation of a Laves phase (NbNiAl) was observed whereas the Cr particles remained stable but were hardened to a remarkable extent due to in-diffusion of Ni and Al. This paper presents the results of three-point bending tests performed on as-HIPed and annealed material at room temperature and elevated temperatures. The yielding behavior at room temperature and the creep behavior at 1200K (927°C) were investigated by compression tests.

INTRODUCTION

Intermetallic aluminides, especially NiAl, are attractive candidate materials for high temperature applications. The main advantages of NiAl are the low density, excellent thermal conductivity and oxidation resistance. In order to improve mechanical properties the effects of Cr and Nb as second phase additions were investigated.

EXPERIMENTAL PROCEDURE

NiAl/Cr and NiAl/Nb composite material was prepared by blending gas-atomized NiAl powder with Cr or Nb powder. The content of the second phases varied between 5 and 10 weight percent (in volume percent: 4.1 - 8.3 for Cr; 3.4 - 7 for Nb). In the case of Cr, powders with two different particle fractions have been used. Typical impurity concentrations and average particle sizes of the powders are given in Table I.

Table I: Chemical analysis and average particle size of powders used.

	NiAl	Cr < 63 μm	Cr > 63 μm	Nb
O (ppm)	185	220	200	1000
N (ppm)	10	< 5	< 5	67
H (ppm)	3	3	3	40
average particle size (μm)	73	40	85	17

The blended powders were filled in steel cans (inner diameter: 45 mm; length: 100 mm) which were evacuated, sealed and compacted by hot isostatic pressing (HIP). After the HIP process (3 hours at 1100°C under a pressure of 2000 bar) fully densified material was obtained. The microstructure of NiAl-10 wt%Cr and NiAl-10wt%Nb is shown in Fig. 1a and b.

The mechanical properties of the NiAl matrix and the composite materials as well the alteration of these properties due to annealing at 1473K (1200°C) were measured by means of three-point bending tests and compression tests. Three-point bending tests were performed at room temperature and temperatures between 773K (500°C) and 1373K (1100°C). From these

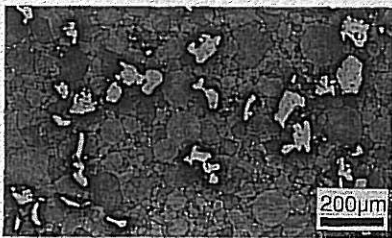


Fig. 1a: NiAl-10wt%Cr after HIPing.

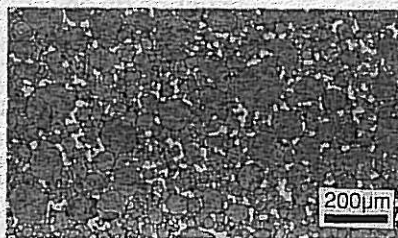


Fig. 1b: NiAl-10wt%Nb after HIPing.

experiments the ductile-to-brittle transition temperatures (DBTT) were determined. The yielding behavior at room temperature (i.e. below DBTT) and the creep behavior at 1200K (i.e. above DBTT) were investigated by strain-rate controlled compression tests on cylindrically shaped samples.

RESULTS AND DISCUSSION

Phase stability

SEM examination of as-HIPed NiAl/Nb material indicated a reaction between NiAl and Nb which leads to the formation of additional phases. Fig.2 shows a SEM micrograph of a Nb particle which is covered by two phases. From XRD measurements the phase in contact with the NiAl matrix was identified as NbNiAl (Laves type, C14 structure), whereas the inner phase was referred to Nb₂NiAl [1,2]. Due to the formation of the NbNiAl phase the hardness in the reaction zone is increased to a threefold value as compared to the NiAl matrix.

In the case of as-HIPed NiAl/Cr material no indication about the presence of additional phases was found by XRD and SEM. However, due to diffusion of Ni and Al an increase in hardness was measured in the Cr particles near the NiAl/Cr interface.

In order to investigate the thermal stability of Nb and Cr within the NiAl matrix at elevated temperatures and longer durations, samples were annealed under vacuum conditions

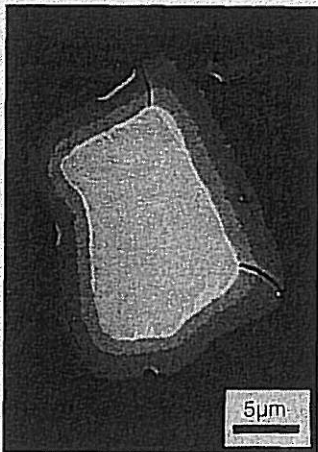


Fig. 2: Backscatter electron micrograph of Nb particle after HIP process.

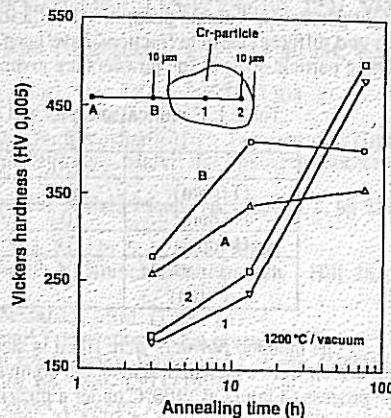


Fig. 3: Room temperature hardness of Cr particles and NiAl matrix as a function of annealing time (annealing temperature: 1200°C)



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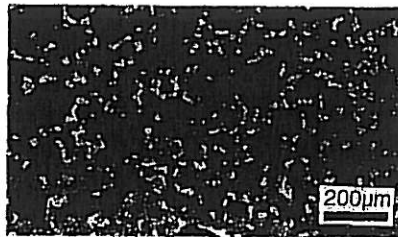


Fig. 1b: NiAl-10wt%Nb after HIPing.

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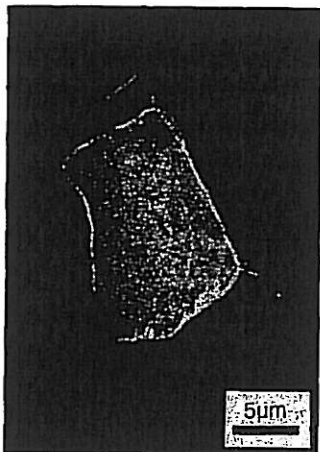


Fig.2: Backscatter electron micrograph of Nb particle after HIP process.

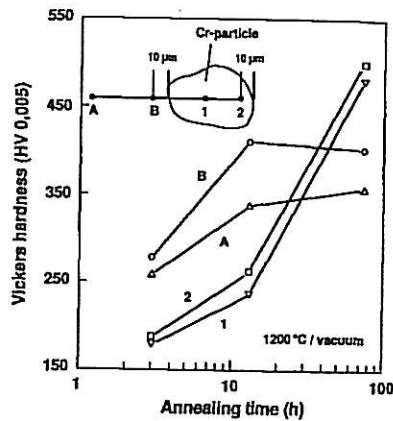


Fig.3: Room temperature hardness of Cr particles and NiAl matrix as a function of annealing time (annealing temperature: 1200°C)

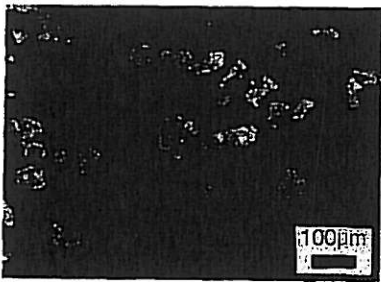


Fig.4a: SEM micrograph of NiAl-10wt%Cr after densification.

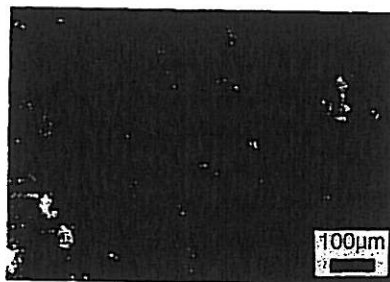


Fig.4b: SEM micrograph of NiAl-10wt%Cr after annealing at 1200°C for 70 hours.

at 1200°C for 10 and 70 hours. In the case of NiAl/Nb full transformation of the Nb particles to NbNiAl was observed after an annealing time of 70 hours. Additionally, due to the solubility of Nb in NiAl strengthening of the matrix by solid solution was found.

Whereas Nb exhibited a strong reaction with NiAl at elevated temperatures the Cr particles remained stable but were hardened to a remarkable extent due to further diffusion of Ni and Al. The dependence of room temperature hardness on annealing time for the Cr particles and the NiAl matrix is illustrated in Fig.3. After the HIP process the hardness of Cr is lower than the hardness of the NiAl matrix. After approximately 50 hours annealing time at 1200°C the hardness of the Cr particles surpasses that of the matrix. A further annealing effect is the appearance of pores at the NiAl/Cr interface which is attributed to the Kirkendall mechanism [2]. The size and the density of the pores is dependent on temperature and annealing time. Figs.4a and b show the microstructure of NiAl-10wt%Cr after HIPing (no porosity) and after a 70 hour heat treatment at 1200°C.

Mechanical Properties

The mechanical properties of the monolithic and the composite NiAl materials and the change of these properties due to annealing at 1200°C were investigated by three-point bending tests on plain beams and by compression tests on cylindrically shaped samples. The testing temperatures ranged from room temperature to 1100°C. The three-point bending tests were performed in vacuum whereas the compressive tests were performed in ambient atmosphere.

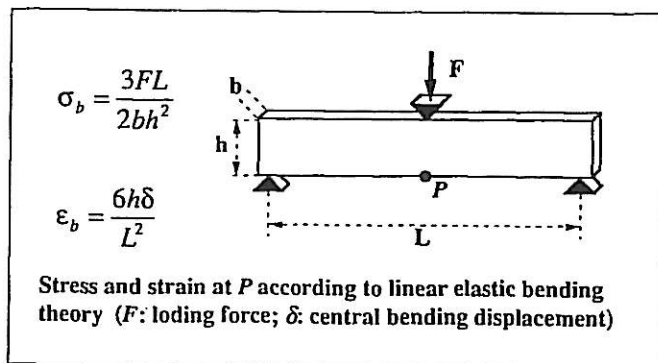


Fig.5: Evaluation of three-point bending tests (see text).

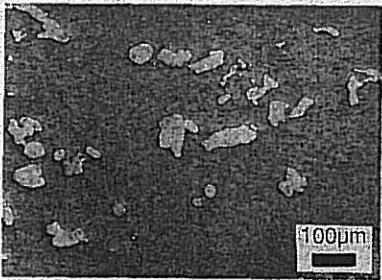


Fig.4a: SEM micrograph of NiAl-10wt%Cr after densification.



Fig.4b: SEM micrograph of NiAl-10wt%Cr after annealing at 1200°C for 70 hours.

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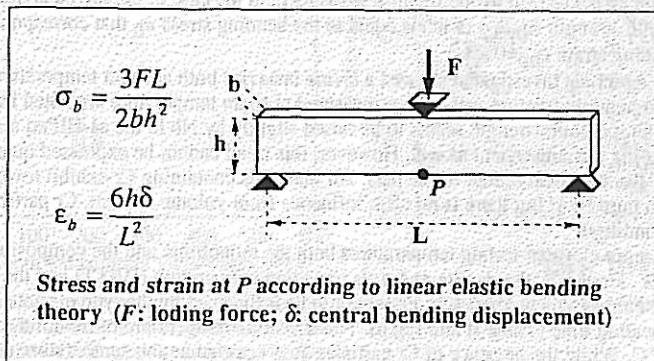


Fig.5: Evaluation of three-point bending tests (see text).

Bending samples of size $5 \times 5 \times 40 \text{ mm}^3$ were prepared via spark erosion. In order to avoid premature fracture caused by surface flaws the samples were polished on the tensile side until roughness values less than $0.1 \mu\text{m}$ were obtained. The span of the three-point bending fixture was 32 mm. The experiments were performed with an Instron type testing machine, the crosshead speed was chosen 0.1 mm/min throughout all tests. During each experiment the central loading point displacement and the load were monitored continuously. Tests were performed at room temperature and at temperatures between 500°C and 1100°C (temperature variation in steps of 50° and 100°). As long as the material behavior is entirely linear-elastic the load displacement curves can easily be transformed to stress-strain curves according to the linear elastic bending theory (see equations in Fig.5). As these equations are no longer valid in the elastic-plastic regime, stresses and strains obtained from non-linear sections of the load-displacement curves are merely effective quantities. These quantities, further referred to with a "b" index, are useful to compare different materials tested in the same way. However, it must always be kept in mind that they are no true stresses and strains, respectively.

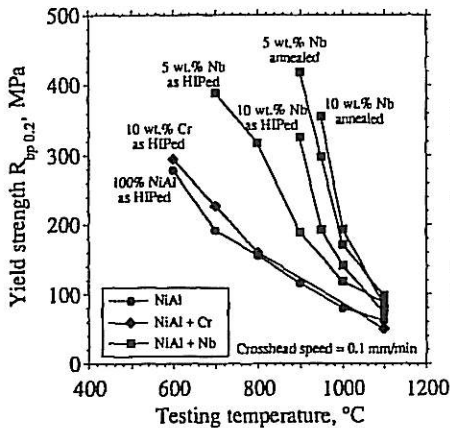


Fig. 6: Bending yield strength $R_{bp0.2}$ as a function of testing temperature.

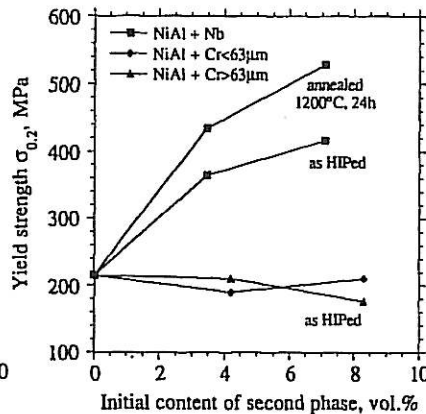


Fig. 7: Room temperature compressive yield stress as a function of initial second phase content.

In this study the material behavior is regarded *brittle* if the plastic bending strain-to-failure is less than 0.2% and *ductile* if this limit is exceeded. In the case of *brittle* fracture the materials are characterized by the fracture stress R_{bF} , in the case of *ductile* behavior by their bending yield strength $R_{bp0.2}$ (which is equal to the bending stress σ_b that corresponds to the plastic bending strain $\epsilon_{bp}=0.2\%$).

All materials investigated showed a *brittle* behavior both at room temperature and at 500°C . The bending fracture values R_{bF} measured at room temperature are listed in Table II. The room temperature strength seems to be raised slightly by Nb in the as-HIPed material and lowered during the subsequent anneal. However, this trend cannot be expressed quantitatively because of the statistical scatter of the data. All materials containing Cr exhibit lower strength values than pure NiAl but there is no clear influence from volume content, Cr particle size or material condition.

At more elevated testing temperatures both the monolithic and the composite materials turn *ductile*. However, the *ductile-to-brittle* transition temperature (DBTT) and the yield stresses just above this temperature turned out to be influenced by the type of second phase and by the annealing time (Table II and Fig.6). The transition temperature of monolithic NiAl is about 500°C . While the presence of Cr particles does not change this temperature considerably, it is raised by Nb. Both the amount of the DBTT shift and the yield stress just above the DBTT increase systematically with Nb content and annealing time.

Table II: Results of three-point-bending tests.

Material	Condition	DBTT (°C)	R _{bF} (MPa)* at room temperature
NiAl	as-HIPed	500-550	459
NiAl-5wt%Nb	as-HIPed	750-800	496
NiAl-5wt%Nb	annealed**	900-950	466
NiAl-10wt%Nb	as-HIPed	900-950	470
NiAl-10wt%Nb	annealed**	950-1000	297
NiAl-5wt%Cr (Cr>63µm)	as-HIPed	500-600	409
NiAl-5wt%Cr (Cr>63µm)	annealed**	600-700	350
NiAl-10wt%Cr (Cr>63µm)	as-HIPed	500-600	411
NiAl-10wt%Cr (Cr>63µm)	annealed**	600-700	404
NiAl-5wt%Cr (Cr<63µm)	as-HIPed	500-600	370
NiAl-5wt%Cr (Cr<63µm)	annealed**	600-700	405
NiAl-10wt%Cr (Cr<63µm)	as-HIPed	500-600	378
NiAl-10wt%Cr (Cr<63µm)	annealed**	600-700	452

*: Mean value of two tested samples

** : Annealing treatment: 1200°C/24h/vacuum

The yielding behavior at room temperature (i.e. below DBTT) and the creep behavior at 1200K (i.e. above DBTT) were investigated by strain-rate controlled compression tests on cylindrically shaped samples of diameter 9 mm and length 18 mm. Experimental details are described in Ref.[3].

Room temperature compression tests were performed at a constant strain rate of $1 \times 10^{-4} \text{ s}^{-1}$. The yield strength was defined to be the compressive stress corresponding to 0.2 % plastic strain. In Fig.7 the results are shown as a function of the initial second phase volume fraction. Again it becomes obvious that the presence of Nb has a much greater influence than that of Cr. However, due to pore formation in the case of Cr no tests on annealed NiAl/Cr material were performed. Moreover, as shown in Fig.8 there is a clear correlation between the room temperature yield strength and the brittle-to-ductile transition temperature.

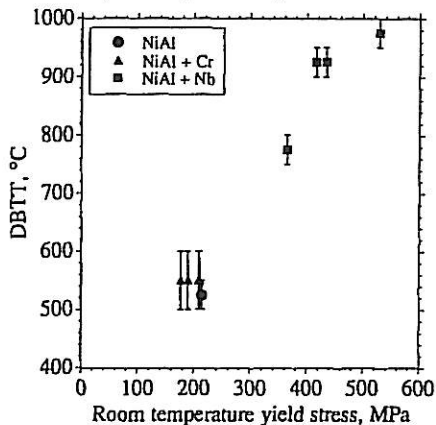


Fig.8: Correlation between compressive yield stress at room temperature and the DBTT measured in bending tests at elevated temperatures.

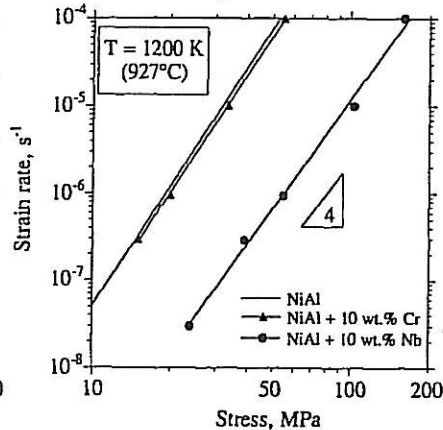


Fig.9: Creep behaviour at 1200K (927°C).

The creep behavior at 1200K (927°C) was investigated by measuring the steady state stress as a function of the applied strain rate, which was varied between $2 \times 10^{-8} \text{s}^{-1}$ and $1 \times 10^{-4} \text{s}^{-1}$. The results obtained for pure NiAl (as HIPed), NiAl-10wt% Cr (as-HIPed) and NiAl-10wt% Nb (annealed) are shown in Fig.9. Due to the increase of the DBTT in the case of NiAl/Nb the creep strength is considerably higher than in the case of monolithic NiAl and NiAl/Cr composite material. This improvement of creep strength is partly caused by the formation of Laves phase and by solid solution strengthening [4]. However, up to now the quantitative contributions of these two mechanisms are not fully understood.

CONCLUSION

NiAl/Nb and NiAl/Cr composite materials were prepared by a powder metallurgical approach. The thermodynamic stability of the second phases at 1200°C was studied. Whereas Nb reacts with NiAl to a ternary Laves phase, Cr shows no reaction with NiAl but is hardened due to interdiffusion of Ni and Al. A further annealing effect is the appearance of pores at the NiAl/Cr interface. The mechanical properties (i.e. DBTT, yield strength, creep behaviour) of the monolithic and composite NiAl materials and the change of these properties due to annealing at 1200°C were investigated by means of three-point bending and compression tests. While Cr particles do not change the DBTT considerably, it is raised in the case of Nb. In addition, a clear correlation between the room temperature yield strength and the DBTT was found. Creep tests at 1200K (927°C) revealed that the NiAl/Nb composites are remarkably stronger than the NiAl/Cr composites and the pure NiAl material investigated.

Future Work

In bending tests performed below the ductile-to-brittle transition temperature both the pure and the composite NiAl materials investigated in this study show only little ductility. The lack of low temperature ductility is regarded to be an obstacle for their use in mechanical applications, even though the problem of brittleness does not exist in the potential service temperature range. In order to examine the brittle behaviour of this class of materials in detail controlled crack growth experiments and tensile tests at room temperature are currently being performed. By optimising the composition and the distribution of the second phase particles it should be possible to raise the fracture toughness and achieve increased ductility which results from multiple microcracking even under tensile loading conditions.

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