

DISPERSION STRENGTHENED INTERMETALLICS BY MECHANICAL ALLOYING: CREEP RESULTS AND DISLOCATION MECHANISMS

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Abstract

In order to increase the creep strength, small dispersoids were introduced into the intermetallic compound NiAl by mechanical alloying. Under favorable conditions, the resulting specific creep rates approach those of superalloys. To accompany these experimental effects, a new model has been developed which predicts tendencies for the success of dispersion strengthening in ordered alloys. It is shown that the dissociation distance of the partial dislocations, relative to the dispersoid particle size, has an important effect on the strengthening achievable.

Introduction

The creep strength of monolithic intermetallics such as aluminides is typically by far inferior to that of advanced superalloys [1]. Recent developments have sought to improve the high temperature strength of NiAl by preparing particulate composite materials containing second phase particles of more than 10 vol.% [2]. The probably first study on oxide dispersion strengthened (ODS) intermetallic alloys [3] showed the producibility, in principles of ODS-NiAl and ODS-FeAl based on a processing technique that is now called Mechanical Alloying (MA). The main advantage of ODS alloys lies in the retention of useful creep strength up to high homologous temperatures, where other strengthening mechanisms are no longer effective [4]. Model approaches of creep in dispersion strengthened, disordered alloys show that the choice of the dispersoid parameters has a strong influence on the efficacy of dispersion strengthening [4, 5]. However, no real theoretical understanding of creep in dispersion strengthened ordered alloys exists [6] and thus, the promises and limitations of dispersion strengthened intermetallics are unclear. The present paper addresses new experimental and theoretical aspects of this promising new class of high-temperature materials.

Experimental

Processing of dispersion strengthened and composite materials

ODS NiAl materials were prepared from prealloyed, gas-atomised Ni₃Al and Al-24 at.% Ni powders supplied by Homogeneous Metals, Inc.. Mechanical Alloying was carried out with 1 to 2 vol.% Y₂O₃ dispersoid powder for 50 hours in a high energy ball mill at the Metallgesellschaft AG Frankfurt. Milled powders were vacuum canned in stainless steel and hot extruded at 1200°C [7]. Further details can be found in table 1.

The preparation of NiAl-NiAlNb composite materials is described elsewhere in these proceedings [8]; it resulted in globular second phase particles, randomly distributed in the NiAl matrix, with a mean diameter of 18 µm. As an alternative to the cryomilling process [2], a NiAl alloy containing about 10 Vol.% AlN was prepared by a gas-metal absorption reaction: atomised NiAl powder was milled for 1h under argon to an average particle size of about 7 µm; the as milled powder was heat treated at 1200°C in a nitrogen atmosphere with a partial N₂-pressure of 300 hPa. X-ray diffraction analysis confirmed the formation of AlN after heat treatment. The nitrogen concentration was determined by WDS analysis (see table 1). The resulting AlN volume fraction can be estimated, assuming all nitrogen had reacted to AlN, to be maximum 10 vol.%. Consolidation was performed by HIP [8]. The formation of AlN took place at the NiAl-powder surfaces. In the bulk material, regions near grain boundaries show high amounts of fine AlN dispersoids (mean diameter about 50 nm) while grains are mostly free of AlN.

Creep tests

Cylindrical compression specimens were electrodischarge machined from the compacted materials. Constant strain rate compression tests ranging from 10^{-4} to 10^{-8} s^{-1} were conducted at 1200 to 1300 K in a universal testing machine to about 5% plastic strain. The recorded stress-strain data were converted to true values. In every case a steady state creep behaviour was observed. After the test, the specimens were cooled down under the current steady state stress to avoid any relaxation of dislocation structures.

TEM investigations

TEM investigations were carried out on sections perpendicular to the extrusion axis. Specimens were prepared by a combination of electrodischarge machining (250 μm thick), soft mechanical grinding (100 μm , \varnothing 3 mm) and dimpling to 20 μm , followed by ion milling.

Alloy	Composition	Consolidation	Heat treatment	Matrix grain size
IP 6000	Ni-50 at.%Al + 1 vol.% Y_2O_3	hot extrusion	-	1 μm
IP 6000 A	Ni-50 at.%Al + 1 vol.% Y_2O_3	hot extrusion	1400°C/10h	10 μm
IP 6002	Ni-50 at.%Al + 2 vol.% Y_2O_3	hot extrusion	-	< 1 μm
ODS-Fe40Al [9]	Fe 40 at.% Al + 2 vol.% Y_2O_3	HIP	-	15 μm
NiAl/NiAlNb	Ni-50 at.%Al + 10 wt.% Nb	HIP	1200°C/12h	36 μm
NiAl/AlN	Ni-50 at.% Al + 2 wt.% N	HIP	-	10 μm

Table 1: Chemical composition, processing parameters and grain sizes

Microstructure of ODS-Aluminides

The matrix grain size was found to lie in the micrometer range as is common after mechanical alloying (see IP6000 and IP6002 in table 1). The dispersoids, of mean size 40 nm, are homogeneously distributed with a mean free spacing of about 300 nm. Only in alloys with coarser grains, realized by heat treatment, we did find dislocation configurations as in Fig. 1. These configurations suggest an interaction between dislocations and the yttria-dispersoids in both alloys, as expected for ODS materials [10, 11].

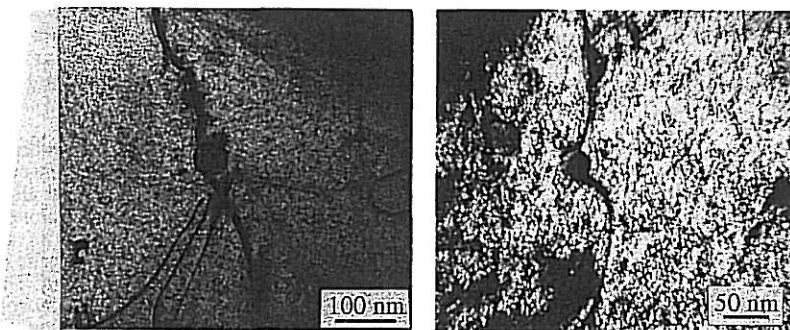


Fig. 1: Attractive interaction between a dispersoid and a dislocation in ODS-Fe40Al (left) and IP6000A (right) tested at moderate creep rates and 1200K and 1300K resp. Dispersoid diameter in both configurations $d=40 \text{ nm}$. The dislocations are bowed out in their glide directions.

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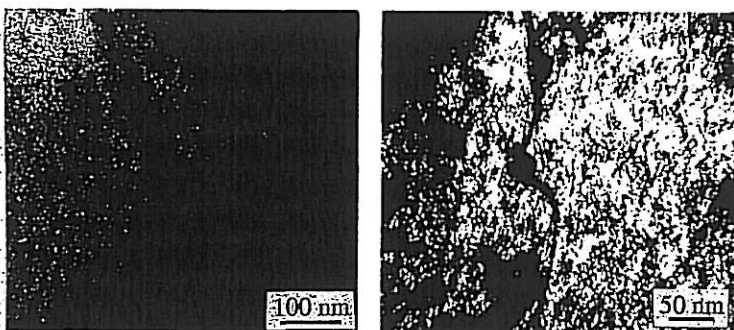


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Creep strength of ODS - Aluminides and NiAl composite materials

A high stress exponent ($n=27$), typical for ODS materials, could be observed for IP6000 A at test temperatures of 1200 and 1300 K (see Fig. 2). The grain size of this material could be increased during a heat treatment (see table 1). Increasing the dispersoid volume fraction from 1 to 2 Vol.% resulted in a higher stress level at 1200 K and a higher stress exponent in the fine grained, as extruded material IP6002 (see Fig. 3). Attempts to coarsen grain sizes in IP6002 were not yet successful. The ODS-NiAl materials investigated show a clearly higher creep resistance at 1200K than ODS-Fe40Al. The NiAl/AlN composite material exhibits a higher creep resistance than NiAl/NiAlNb with comparable second phase volume fractions, mainly due

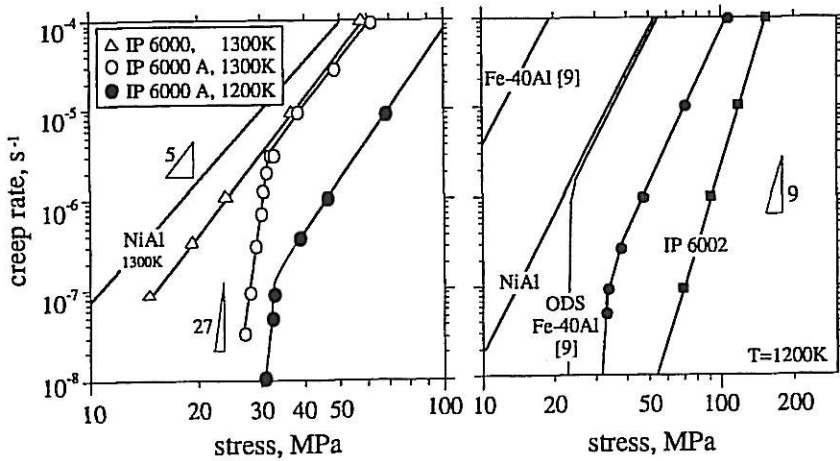


Fig. 2. Effect of oxide dispersion strengthening, grain size and test temperature on creep strength of NiAl

Fig. 3. Influence of dispersoid volume fraction on creep strength of NiAl in comparison to ODS-FeAl [9].

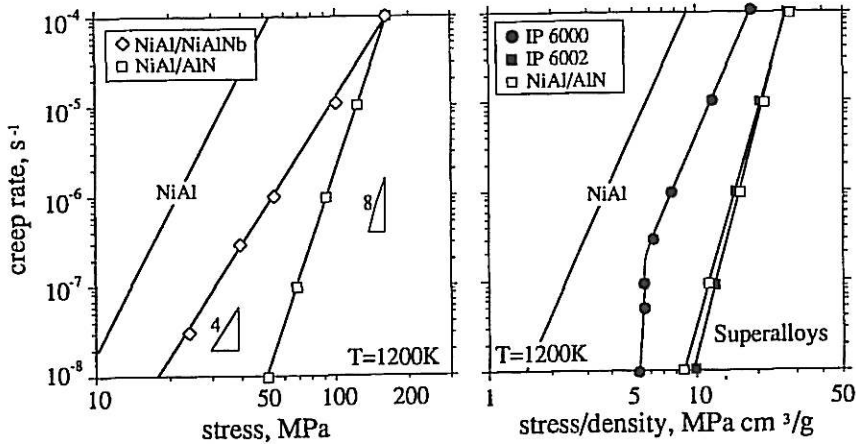


Fig. 4. Creep strength of two particulate composite materials, tested at 1200 K in air.

Fig. 5. Density compensated comparison of advanced NiAl materials with superalloys at 1200 K

to a higher stress exponent (Fig. 4). In a density compensated point of view, ODS NiAl materials reach the creep resistance of some superalloys at 1200 K, especially at low strain rates. Note that dispersion strengthened, but still fine grained IP6002 nearly shows the same creep behaviour at 1200 K as the NiAl/AlN composite material.

Modelling of Dispersion Strengthening in Ordered Alloys: First Results

The theory of dispersion strengthening at high temperatures has been extended considerably in recent years. The main result is that the effect of creep retardation can be explained if an attractive dispersoid-dislocation interaction exists (for details see [4, 5, 12]). A consequence of this effect is that a dislocation which climbs over a dispersoid meets two "obstacles" (fig. 6): one is due to the necessary increase in line length during climb, the other to the barrier to detachment from the dispersoid. An important parameter for the creep strength is the athermal detachment stress τ_d , which is given by [12]

$$\tau_d = \tau_0 \sqrt{1-k^2}$$

where τ_0 is the Orowan shear stress, and k a factor measuring the strength of the attractive interaction.

This model has recently been extended to ordered matrix materials (see [15]). The major modification is due to the dissociation of dislocations into superpartials which have now to be taken into account (fig. 7): because the partials experience different back stresses (as shown in fig. 6) and because they interact elastically, the detachment stress - and thus the creep strength - now depend on the dissociation spacing relative to the dispersoid size. The simple analysis in [15] shows that the detachment stress for the leading partial τ_d is given by the arithmetic mean of its own back stress $\tau(d)$ and that on the trailing partial $\tau(d-w)$:

$$\tau_d = \frac{\tau(d) + \tau(d-w)}{2}$$

where w is the spacing of the superpartials.

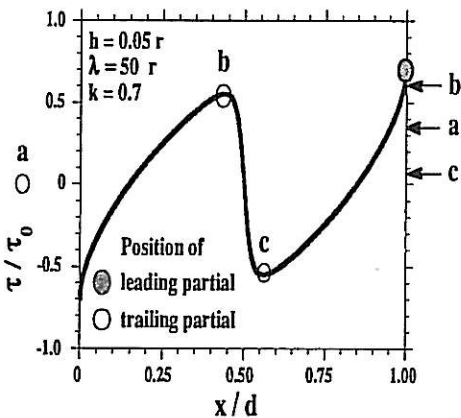


Fig. 6. Back stress - distance profile as calculated in [12] for a single dislocation. The effect of the spacing w between superpartials on the detachment stress of the leading partial: a) $w > d$ (dispersoid diameter), b) $w \approx 0.55 d$, c) $w \approx 0.45 d$. The arrows indicate the resulting stress level for detachment in the three cases.

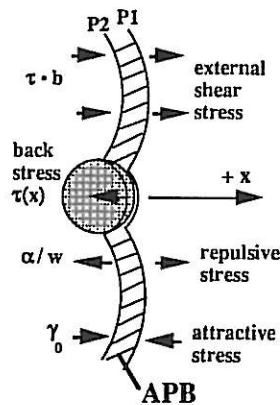


Fig. 7. Dispersoid-dislocation interaction in an ordered matrix. The superpartial spacing is fixed by mutual forces on the superpartials.

Fig. 6 shows in three special cases the change in the detachment stress of a leading partial as a function of the separation width of the superdislocations:

Case a) The detachment stress is reduced to half the value of a single dislocation if the partial spacing is greater than the dispersoid diameter (leading partial at $x=d$, trailing partial at "a").

Case b) The detachment stress of an extended superdislocation is relatively high if the trailing partial sits at the point of the climb barrier maximum (at "b").

Case c) The detachment stress decreases to even negative values, if the trailing partial resides in the valley of the stress profile ("c") The trailing partial facilitates the detachment process for the leading partial

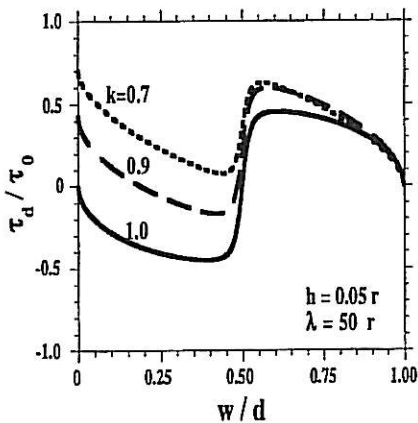


Fig. 8. The detachment stress for the leading partial under the influence of the trailing partial, as a function of the relation partial spacing to dispersoid diameter.

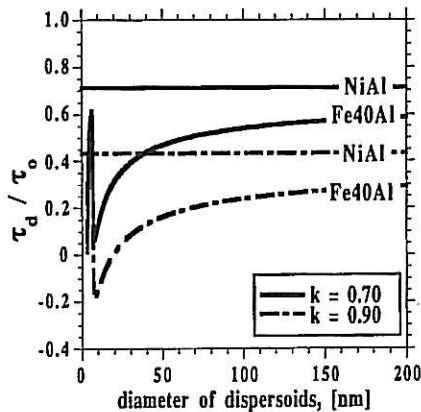


Fig. 9. Athermal detachment stress of the leading partial for NiAl with $\gamma = 810 \text{ mJ/m}^2$ [13] and $w=0$ and Fe-40at%Al with $\gamma = 150 \text{ mJ/m}^2$ and $w = 7 \text{ nm}$ (Data extrapolated from [14]) for different values of relaxation parameters k .

The detachment stress of the leading partial is now a function of the relation partial spacing to dispersoid diameter (Fig. 8). An undissociated superdislocation has the maximum detachment stress ($w/d = 0$) whereas an extended superdislocation has two possibilities to achieve a high detachment stress: i) to approach the undissociated case, i.e. $w/d = 0$ or ii) $w/d = 0.55$. The second possibility dominates over the first one if the attractive interaction is small ($k \approx 1$). For usual applications ($0.7 < k < 1.0$) the maximum variation in detachment stress depending on the relation w/d can be considerable. For further details the reader is referred to [15].

A first application of these results to intermetallics is shown in Fig. 9. For Fe-40at%Al and NiAl we can estimate the optimum dispersoid sizes to achieve an appreciable detachment stress. It is seen that the athermal detachment barrier in NiAl is practically independent of dispersoid size whereas in Fe-40at%Al the detachment barrier increases with dispersoid size. Therefore it is expected that NiAl would be more responsive to strengthening by small dispersoid particles than FeAl; this is in qualitative agreement with our experiments.

Conclusion

Dispersion strengthened aluminides can successfully be prepared by Mechanical Alloying. A high stress exponent, which is typical for the creep behaviour of dispersion strengthened materials, can be observed for NiAl and FeAl if the grain structure is coarsened after consolidation. An improvement in creep properties can be obtained even by moderate grain growth. A further increase in creep strength is attained by an optimised dispersoid volume fraction. Particulate composite materials also improve the creep properties of NiAl. A new processing technique leads to NiAl/AlN composite materials with controllable grain sizes and second phase volume fractions. Our results indicate that a finely dispersed AlN composite material is preferable to a coarser second phase NiAl/NiAlNb material, due to a higher stress exponent.

TEM investigations of ODS aluminides suggest that the mechanism of dispersion strengthening in ordered alloys is similar to that in disordered alloys but is affected by the dissociation of dislocations into superpartials. Our new model takes this effect into account. It is concluded that high creep strength of ordered dispersion-strengthened materials requires an optimum relation between the partial spacing, which is determined by the APB energy, and the dispersoid size. Alloy development along this line is currently in progress.

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