



Short communication

## Enhancement of fracture toughness of hot-pressed NiAl-Re material by aluminum oxide addition

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## ABSTRACT

In the search for a remedy to increase the fracture toughness of NiAl, the effect of rhenium and aluminum oxide addition is explored. Using a powder metallurgy processing route an optimum composition of NiAl-Re-Al<sub>2</sub>O<sub>3</sub> material is found which manifests  $K_{IC}$  over two times higher than as-received NiAl sintered under the same conditions.

### 1. Introduction

Research efforts aimed at improving the mechanical properties of nickel aluminide (NiAl) at room temperature (RT) to make it applicable in bulk aerospace parts have been continued for more than 40 years now [1–3]. The progress in enhancing NiAl properties is significant [4], but the low fracture toughness at RT still remains an unresolved issue. Among other factors limiting the applicability of NiAl, an insufficient ductility [5] and lack of impact resistance [6] should be mentioned. On the other hand, high-temperature properties of NiAl such as thermal conductivity of 76 W/mK, melting point of 1676 °C [7], and good oxidation resistance [8] fully satisfy the industrial requirements.

It was shown in Ref. [9] that a small addition of rhenium improved significantly the fracture toughness of NiAl fabricated by powder metallurgy method. The selection of rhenium, which is a refractory metal, was motivated by the envisaged applications of NiAl in high temperature regimes. For 1.25 at.% (1.53 vol%) of rhenium, which corresponds to its eutectic concentration in NiAl-Re alloys obtained by directional solidification [10], the fracture toughness of the hot-pressed NiAl 1.25Re material obtained in Ref. [9] increased to 12.6 MPa√m as compared with 7.2 MPa√m for the as-received NiAl sintered under the same conditions. However, this gain in fracture toughness was still unsatisfactory from the application point of view.

In this short communication a further improvement of the NiAl fracture toughness at RT is sought through (i) addition of alumina ceramic particles to NiAl-Re powder mixture, and (ii) adjustment of the

milling process conditions.

A beneficial effect of Al<sub>2</sub>O<sub>3</sub> on the mechanical properties of NiAl was reported in the past [11,12]. However, in research papers devoted to enhancing the mechanical properties of nickel aluminides (see Ref. [4, 13] and references therein) a combined effect of rhenium and aluminum oxide additions on NiAl properties was not addressed. When a ceramic phase is added to NiAl the processing routes other than commonly used casting techniques should be considered in order to avoid segregation of the ceramic particles. To this end, powder metallurgy (PM) is one of the reasonable choices [14–16]. Incidentally, the PM was successfully implemented to produce TiAl turbine blades as an alternative technique to directional solidification [17].

Besides fracture toughness, which is the central material property of this investigation, other mechanical properties such as bending strength, tensile strength and ductility need to be controlled. The ductility of polycrystalline NiAl at room temperature is relatively low [5]. Ramasundaram et al. [18] have found that in NiAl–Mo composites the increasing fracture toughness at RT was accompanied by a decreasing ductility. According to Pope and Darolia [19] a minimum of 1–2% strain at rupture is required to sustain the stresses that can be generated during machining and assembly of engine components.

The primary aim of this study is to show how the addition of α-Al<sub>2</sub>O<sub>3</sub> ceramic particles to NiAl-Re powder mixture affects the fracture toughness of NiAl-Re-Al<sub>2</sub>O<sub>3</sub> composite fabricated by pressure-sintering (HP). The secondary aim is to explore how the milling time and protective milling conditions influence the mechanical properties of the

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**Table 1**

Selected results from particle size analysis. Abbreviations (4h), (8h) denote milling times of 4 and 8 h in air, respectively; (4h in heptane) stands for 4 h milling in heptane. NiAl 0.73Re 0.5Al<sub>2</sub>O<sub>3</sub> (8h + 4h) means that NiAl powder was first milled for 8 h in air, then rhenium and aluminum oxide were added and the resulting powder mixture was further milled in air for 4 h.

Material	Particle size [μm]		
	Dv10	Dv50	Dv90
NiAl (Goodfellow)	11.80	35.80	96.00
Rhenium (Ecoren)	32.30	82.10	206.00
Al <sub>2</sub> O <sub>3</sub> (NewMetKoch)	0.54	1.12	2.62
NiAl (4h)	2.23	7.19	16.80
NiAl (8h)	2.18	6.56	14.20
NiAl (4h in heptane)	1.20	3.46	6.24
NiAl 0.73Re 0.5Al <sub>2</sub> O <sub>3</sub> (8h + 4h)	1.65	5.29	10.50

NiAl-based materials under study. Finally, it will be checked whether an improvement of fracture toughness of this new material is accompanied by an enhancement of tensile ductility.

## 2. Materials and methods

A commercial NiAl powder (GoodFellow, 99.0% purity, mean particle size 35.8 μm) was used to obtain NiAl/Re/Al<sub>2</sub>O<sub>3</sub> powder mixtures in a planetary ball mill. The admixture of rhenium (Metraco, 99.0% purity, mean particle size 82.1 μm) was in the range of 0.6–1.5 at.%, which corresponds to 0.73–1.83 vol%. The volume fraction of aluminum oxide α-Al<sub>2</sub>O<sub>3</sub> (NewMetKoch, 99.99% purity, mean particle size 1.1 μm) was 0.5, 1, 2, and 10 vol%. A high energy ball mill (Fritsch Pulverisette 5) was employed to prepare powder mixtures (200 rpm, ball to powder weight ratio BPR 5:1, 20 min break after each 10 min of milling). The milling times for all the materials fabricated in this study are given in the next section. Throughout the paper the admixtures of Re and Al<sub>2</sub>O<sub>3</sub> are given in vol%.

Previous research [9] has shown that during milling of NiAl in air some aluminum oxide is formed *in situ*. To control this effect alternative milling conditions were adopted including a special milling chamber, heptane as the liquid medium and inert gas (argon) as the protective atmosphere. The milling in heptane was carried out for selected material compositions with starting powders being placed simultaneously in the chamber without initial milling of pure NiAl powder. Before start of milling in heptane the chamber was purged with argon. A particle size analysis was performed using Malvern Mastersizer 3000.

To find the optimum composition of NiAl-Re-Al<sub>2</sub>O<sub>3</sub> material a number of different powder mixtures were sintered in a hot press (HP, Thermal Technology LLC) in argon atmosphere. The sintering parameters were: temperature 1400 °C, pressure 30 MPa, holding time 60 min. The heating rate was 10 °C/min. The sample dimensions after removal from the furnace were 48 mm in diameter and about 5–10 mm in height.

The microstructure was characterized by scanning electron microscopy (Zeiss Crossbeam 350). The fracture toughness in SEVNB (Single-edge V-notch beam) probe was performed on prismatic specimens (3 × 4 × 25 mm<sup>3</sup>) according to ISO 23146 [20]. The bending strength was measured on unnotched prismatic specimens (3 × 4 × 25 mm<sup>3</sup>) according to ISO 14704 [21]. Both types of tests were run in four point bending mode on a Zwick ProLine Z50 Universal Testing Machine. Tensile tests were performed on MTS 858 hydraulic machine on non-standard flat dog-bone specimens (see Fig. 4a for specimen dimensions) at a displacement rate of 0.15 mm/min, which corresponds to the average strain rate of 3 · 10<sup>-4</sup> s<sup>-1</sup>. The strain was determined by digital image correlation (DIC) according to the procedure described in Ref. [22].

The specimens for bending strength, fracture toughness and tensile tests were prepared by wire-cutting (Mitsubishi MV1200R). All the flat faces were ground on a diamond disc using polishing clothes with diamond suspension up to 1 μm (Presi Mecatech 334). The V-notches on

**Table 2**

Mechanical properties of NiAl-based compacts with admixture of rhenium and aluminum oxide (in vol%). All tests were performed at room temperature. Notations in round brackets were explained in Table 1 caption. The best material is marked in bold face.

Material	Relative density [%]	Fracture toughness [MPa√m]	Bending strength [MPa]
1. NiAl (as received [12])	99.50	7.20 ± 0.40	345.6 ± 53.8
2. NiAl (4h)	99.74	8.08 ± 0.53	428.9 ± 71.8
3. NiAl (8h)	99.59	9.08 ± 1.03	856.1 ± 129.1
4. NiAl (4h in heptane)	99.10	12.39 ± 2.05	869.0 ± 138.6
5. NiAl 1.53Re (4h in heptane)	99.69	12.61 ± 1.33	855.5 ± 160.2
<b>6. NiAl 0.73Re 0.5Al<sub>2</sub>O<sub>3</sub> (8h + 4h)</b>	<b>99.87</b>	<b>15.19 ± 2.51</b>	<b>1065.1 ± 48.2</b>
7. NiAl 0.73Re 0.5Al <sub>2</sub> O <sub>3</sub> (4h in heptane)	99.52	13.57 ± 2.02	920.8 ± 89.8
8. NiAl 1.53Re 0.5Al <sub>2</sub> O <sub>3</sub> (8h + 4h)	99.86	13.73 ± 1.46	978.3 ± 87.4
9. NiAl 1.83Re 0.5Al <sub>2</sub> O <sub>3</sub> (8h + 4h)	99.22	11.30 ± 1.69	720.6 ± 46.2
10. NiAl 0.73Re 1Al <sub>2</sub> O <sub>3</sub> (8h + 4h)	99.75	13.89 ± 2.42	991.5 ± 100.3
11. NiAl 0.73Re 1Al <sub>2</sub> O <sub>3</sub> (4h in heptane)	99.83	11.18 ± 0.79	963.6 ± 102.5
12. NiAl 1.53Re 1Al <sub>2</sub> O <sub>3</sub> (8h + 4h)	99.68	13.22 ± 2.11	876.2 ± 92.6
13. NiAl 1.83Re 1Al <sub>2</sub> O <sub>3</sub> (8h + 4h)	99.30	11.26 ± 2.30	1024.8 ± 108.8
14. NiAl 1.53Re 2Al <sub>2</sub> O <sub>3</sub> (8h + 4h)	99.72	12.27 ± 2.30	736.4 ± 89.0
15. NiAl 1.53Re 10Al <sub>2</sub> O <sub>3</sub> (8h+4h)	98.80	11.59 ± 1.77	857.96 ± 72.7

SEVNB specimens were prepared using a custom-made notching machine with a razor blade and a diamond paste (9 and 6 μm).

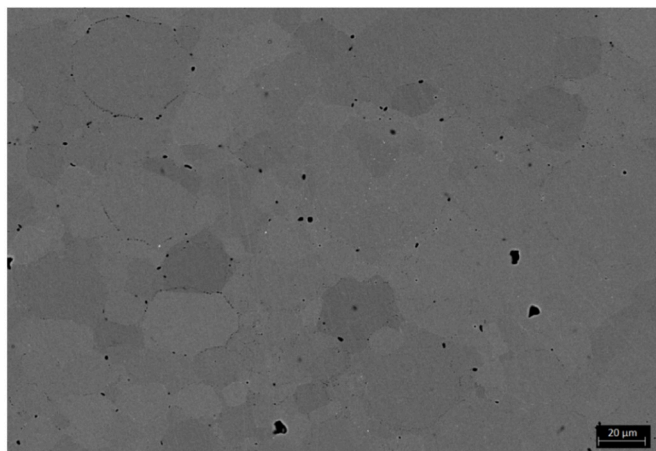
## 3. Results and discussion

The results of particle size analysis are presented in Table 1. The first three lines correspond to as-received starting powders. After 4 h of milling in air the average particle size of pure NiAl powder, denoted as NiAl (4h), was reduced from 35.80 μm to 7.19 μm. It was only slightly bigger than the average particle size (6.56 μm) of NiAl milled for 8 h in air (NiAl (8h)). When NiAl was milled for 4 h in heptane (NiAl (4h in heptane)) the average particle size (3.46 μm) was significantly smaller than that of NiAl (4h). In the case of NiAl milled for 8 h in air and then for further 4 h with admixtures of rhenium and alumina (NiAl 0.73Re 0.5Al<sub>2</sub>O<sub>3</sub> (8h + 4h)) in air the average particle size was 5.29 μm. The effects of particle size and milling in heptane on the mechanical properties under investigation will be discussed later in this section.

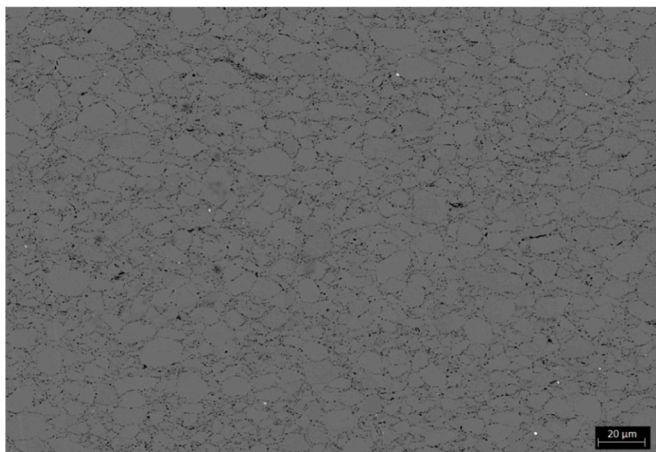
The relative density, fracture toughness and bending strength of the materials produced in this study are displayed in Table 2. As it was shown in Ref. [9] the largest improvement of K<sub>IC</sub> of pressure-sintered NiAl-Re material was obtained for 1.25 at% (1.53 vol%) Re. According to Ref. [10] this is the eutectic concentration of Re in NiAl-Re alloys at 1668 °C. In the present study small amounts of α-Al<sub>2</sub>O<sub>3</sub> were milled together with NiAl and Re powders. Interestingly, the highest K<sub>IC</sub> values in Table 2 are not exhibited by the sintered compacts with the eutectic concentration of rhenium (NiAl 1.53Re). Of all the material variants produced the maximum fracture toughness of 15.19 MPa√m was achieved for NiAl 0.73Re 0.5Al<sub>2</sub>O<sub>3</sub> (8h + 4h) composite, in which the Re content is less than half of the eutectic concentration.

For the NiAl powders sintered without any admixture (upper part of Table 2) the bending strength and fracture toughness increased with the grain size reduction in accordance with the Hall-Petch mechanism as compared with the as-received NiAl powder.

The average grain size of sintered NiAl material is related to the



(a)



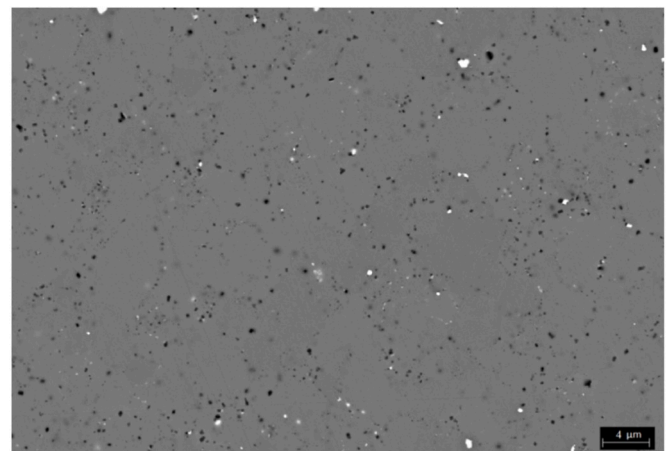
(b)

**Fig. 1.** SEM micrographs of sintered compacts of pure NiAl: (a) as-received; (b) milled 8 h in air NiAl (8h). Sporadic black spots on grain boundaries in image (b) represent alumina formed *in situ* during milling in air.

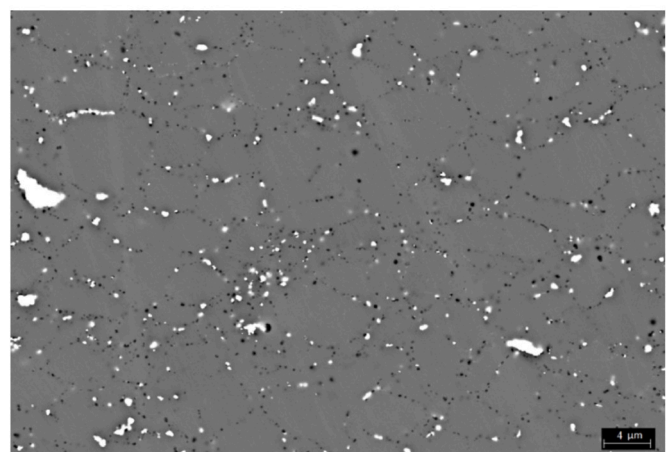
average particle size of starting powder. This can be seen in Fig. 1 where the average grain size of the compact obtained from the as-received NiAl powder (Fig. 1a) is visibly larger than that of NiAl (8h) sintered with finer NiAl particles due to 8 h of milling (Fig. 1b). The size reduction of NiAl particles after milling in heptane (cf. Table 1) may be the reason for higher mechanical properties manifested by NiAl (4h in heptane) (Table 2).

The results for NiAl-Re containing Al<sub>2</sub>O<sub>3</sub> (compositions 6–15 in Table 2) reveal that (1) aluminum oxide addition in the range of 0.5 - 1 vol% generally enhances the fracture toughness and bending strength of NiAl-Re, (2) the optimum composition involves a sub-eutectic concentration of Re (0.73 vol%) and a relatively small amount of alumina (0.5 vol%). A similar reinforcing effect caused by alumina particles when added to a metal matrix was traced back to the good cohesive properties of metal/ceramic interfaces [23].

It was found in Ref. [9] that even if no alumina was added to NiAl-Re powder mixture, some *in situ* formed aluminum oxide was detected by SEM at the grain boundaries due to milling in air. These alumina particles may act similarly as the oxide dispersive strengthening mechanism (ODS) and may also contribute to the fracture toughness of NiAl and NiAl-Re materials. The ODS is a known technique to enhance properties of nickel-based superalloys [24], but it was also successfully used for nickel aluminide [25]. To avoid the formation of *in situ* aluminum oxide some protective measures (argon + heptane) were applied during the milling process. These measures reduced the amount of *in situ* alumina



(a)



(b)

**Fig. 2.** SEM images of sintered NiAl samples made with powders milled under protective conditions (heptane) without Re and with Re addition: (a) pure NiAl (4h in heptane); (b) NiAl 1.53Re (4h in heptane). White spots were identified respectively as: WC from frictional wear of milling balls and chamber (a), Re grains with no traces of WC (b).

but they did not prevent its formation completely.

It was expected that higher reduction of particle size when milled in heptane would enhance the mechanical properties of sintered samples (according to Hall-Petch mechanism). However, it was true only for pure NiAl. When rhenium and ceramic phase were added and milled with NiAl in heptane the mechanical properties of sintered samples were actually lower (see Table 2). This outcome undermines the positive role of milling in heptane in the present work and points to the advantageous effect of milling in air on the mechanical properties of NiAl via the *in-situ* formed alumina.

The EDS spectroscopy revealed also some tungsten carbide on the grain boundaries of NiAl (4h in heptane). Its appearance can be traced back to the gradual wear of tungsten carbide milling balls and the chamber during high energy ball milling. Similar effect was reported in Ref. [26]. The presence of WC was only detected on samples without rhenium, i.e. NiAl (8h) and NiAl (4h in heptane), Fig. 2a (white spots). Hence, it can be inferred that rhenium being a very plastic material acts as a lubricant during high energy ball milling preventing the wear of tungsten carbide balls and chamber. The main reinforcing factor in sintered pure NiAl materials is the grain size reduction, but the *in situ* alumina particles created during powder milling may also contribute to the fracture toughness and bending strength enhancement (Table 2).

In the case of NiAl 1.53Re (4h in heptane) the rhenium phase

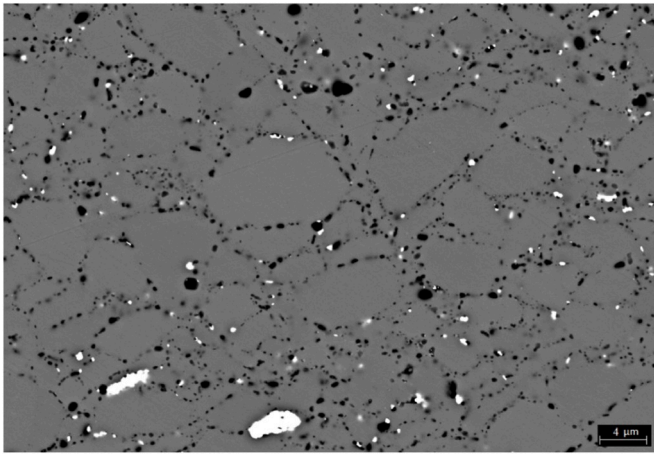


Fig. 3. SEM image of NiAl 0.73Re 0.5Al<sub>2</sub>O<sub>3</sub> (8h + 4h) composite (white spots are Re grains). Small black points on NiAl grain boundaries represent *in situ* formed Al<sub>2</sub>O<sub>3</sub>, whereas larger ones are Al<sub>2</sub>O<sub>3</sub> grains from alumina powder added during material processing.

(Fig. 2b, white spots) is quite homogeneously distributed along the grain boundaries occasionally forming larger grains (>2 μm). They are free from any alloying additions as it was proved in Refs. [9]. No significant differences in grain growth during sintering of NiAl (4h in heptane) and NiAl 1.53Re (4h in heptane) were observed (see Fig. 2a and b) which means that rhenium did not influence this phenomenon.

From the material variants sintered with Al<sub>2</sub>O<sub>3</sub> addition (Table 2) the best performance in terms of toughness and strength is exhibited by NiAl 0.73Re 0.5Al<sub>2</sub>O<sub>3</sub> (8h + 4h) (milled without heptane). Fig. 3 presents a SEM micrograph of the NiAl 0.73Re 0.5Al<sub>2</sub>O<sub>3</sub> (8h + 4h) microstructure. It can be seen that Al<sub>2</sub>O<sub>3</sub> powder added externally leads to bigger alumina grains (above 1 μm) on the NiAl grain boundaries after sintering than that formed *in situ* (nanometric precipitates). The elemental distribution of NiAl 0.73Re 0.5Al<sub>2</sub>O<sub>3</sub> (8h + 4h) revealed no additional phase on the grain boundaries.

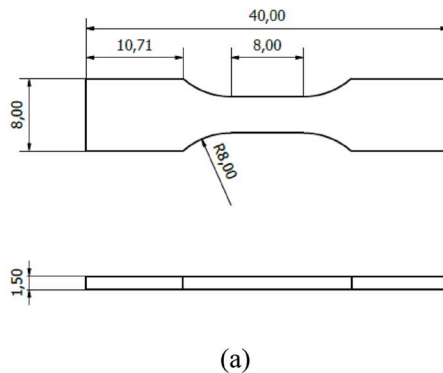
Tensile properties were measured at RT on non-standard flat dog-bone specimens (Fig. 4a) for three selected material compositions: NiAl (as-received, non-milled powder), NiAl (8h) and NiAl 0.73Re 0.5Al<sub>2</sub>O<sub>3</sub> (8h + 4h), the latter exhibiting the highest  $K_{IC}$  (see Table 2). At least 5 specimens were prepared from each material. The results are presented in Table 3.

It is noticeable that longer milling times lead to higher tensile strength values. The optimum admixture of rhenium and aluminum oxide results in a tensile strength enhancement from 338 MPa (pure NiAl as-received) to 605 MPa for NiAl 0.73Re 0.5Al<sub>2</sub>O<sub>3</sub> (8h + 4h), with the strain at rupture reaching almost 0.8%. In comparison with the NiAl–Mo

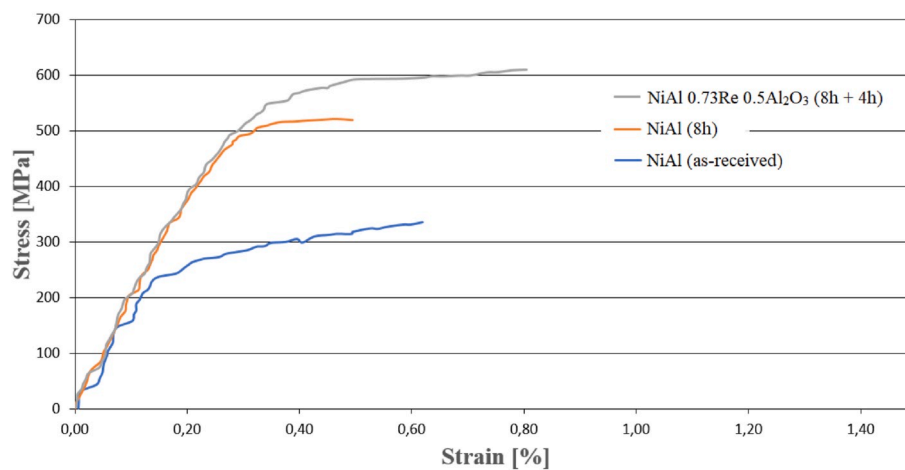
Table 3

Tensile properties of sintered NiAl and NiAl 0.73Re 0.5Al<sub>2</sub>O<sub>3</sub> materials.

Material	Strain at rupture [%]	Tensile strength [MPa]
NiAl (as-received powder)	0.66 ± 0.023	338 ± 2.0
NiAl (8h)	0.52 ± 0.048	520 ± 3.5
NiAl 0.73Re 0.5Al <sub>2</sub> O <sub>3</sub> (8h + 4h)	0.78 ± 0.040	605 ± 4.0



(a)



(b)

Fig. 4. Tensile behavior of pressure-sintered NiAl materials: a) non-standard dog-bone specimen used in uniaxial test (all dimensions in [mm]); b) stress-strain curves for three selected materials.

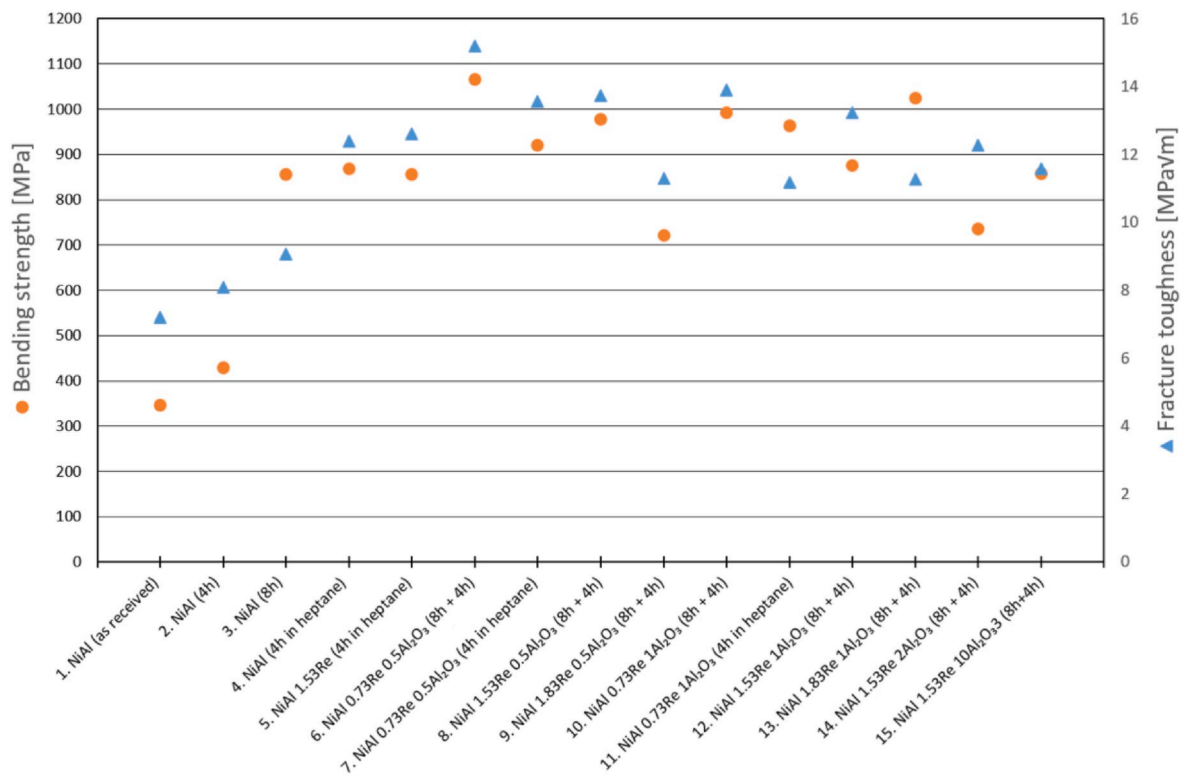


Fig. 5. Overview of strength and toughness data of the materials produced in this research.

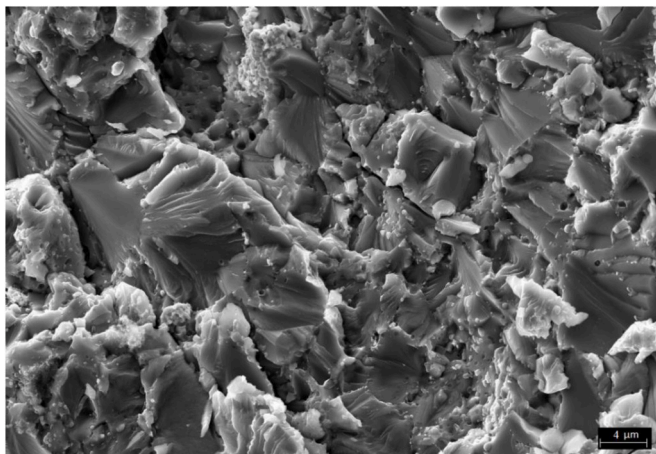


Fig. 6. SEM micrograph of fracture surface of NiAl 0.73Re 0.5Al<sub>2</sub>O<sub>3</sub> (8h + 4h) specimen in tension.

sintered material developed in Ref. [18], which manifested similar fracture toughness ( $\sim 16 \text{ MPa}\sqrt{\text{m}}$ ), the tensile ductility of NiAl 0.73Re 0.5Al<sub>2</sub>O<sub>3</sub> (8h + 4h) (represented here by the rupture strain) and its tensile strength are higher. As remarked in Refs. [18], the inferior tensile properties of NiAl–Mo resulted most likely from negligible plastic deformation of Mo grains prior to fracture.

The stress-strain curves of NiAl materials tested in uniaxial tension (Fig. 4b) manifest similar behavior up to 200 MPa. Above that stress level the as-received NiAl starts to deform plastically, whereas the other two materials remain elastic up to approximately 500 MPa, showing almost identical elastic modulus due to very small amounts of Re and Al<sub>2</sub>O<sub>3</sub> added to NiAl. It is evident that the Re and Al<sub>2</sub>O<sub>3</sub> additions enhance the yield stress (at 0.2% offset) of NiAl, but the grain refinement also plays a role here.

The limited ductility of NiAl (8h) may be attributed to the increasing number of grain boundaries due to milling as well as the *in situ* created aluminum oxide which may hinder plastic deformation. According to Ur et al. [25] in mechanically alloyed NiAl the finely distributed Al<sub>2</sub>O<sub>3</sub> dispersoids may act as significant obstacles to dislocation motion. On the other hand, the addition of rhenium creates coherent interfaces acting as dislocation sources promoting plastic deformation. This is reflected in the highest strain at rupture for NiAl 0.73Re 0.5Al<sub>2</sub>O<sub>3</sub> (8h + 4h). However, the total elongation of 0.78% for this material is still below the acceptable level for structural applications.

In Fig. 5 the bending strength and fracture toughness measurements are collectively shown for all the materials produced in this work. For the majority of the materials (1–11) the strength and toughness values follow the same trend: they either grow or decrease together. However, for the last four materials (12–15), where the additions of rhenium or alumina were essentially higher, this similarity is not preserved.

A typical fracture surface of NiAl 0.73Re 0.5Al<sub>2</sub>O<sub>3</sub> (8h + 4h) specimen shown in Fig. 6 indicates a transgranular type of fracture with crack arrest markings within some cleavage-like facets. This can be interpreted as the crack trapping by regions of higher fracture toughness (e.g. rhenium grains).

#### 4. Conclusions

This paper addresses a recurrent theme in the NiAl development – the improvement of its mechanical properties at room temperature. By introduction of rhenium as a ductile toughening phase and small amount of aluminum oxide to NiAl processed by powder metallurgy, a significant improvement of mechanical properties has been attained. Hot pressing of NiAl–Re–Al<sub>2</sub>O<sub>3</sub> powder mixtures have resulted in final compacts with relative densities above 99%, with the exception of the case with 10 vol% of alumina. A very good homogeneity of the materials has been confirmed during the tensile tests - the standard deviation was less than 0.7%. The best performing material obtained in this work, namely NiAl 0.73Re 0.5Al<sub>2</sub>O<sub>3</sub> manifests the highest improvement of fracture

toughness (15.19 MPa $\sqrt{m}$ ), bending strength (1065.1 MPa), and decent tensile properties (605 MPa rupture stress, 0.8% elongation). However, the tensile ductility is still below 1% and is, thus, insufficient from the application point of view. Future investigations will have to focus on ductility enhancing mechanisms in NiAl 0.73Re 0.5Al<sub>2</sub>O<sub>3</sub> material.

### Originality statement

I write on behalf of myself and all co-authors to confirm that the results reported in the manuscript are original and neither the entire work, nor any of its parts have been previously published. The authors confirm that the article has not been submitted to peer review, nor has been accepted for publishing in another journal. The author(s) confirms that the research in their work is original, and that all the data given in the article are real and authentic. If necessary, the article can be recalled, and errors corrected.

### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

### CRediT authorship contribution statement

**Kamil Bochenek:** Conceptualization, Methodology, Investigation, Writing - original draft. **Witold Węglewski:** Investigation, Writing - review & editing. **Jerzy Morgiel:** Investigation. **Michał Maj:** Investigation. **Michał Basista:** Conceptualization, Methodology, Writing - review & editing, Supervision, Funding acquisition.

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