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Ductilization of Ni_3Al by alloying with boron and zirconium

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ABSTRACT

Purpose: The investigation of Ni_3AlBZr alloys was carried out to determine the influence of small zirconium and boron additions on the microstructure and mechanical properties, particularly with respect to room-temperature, and different strain rate conditions.

Design/methodology/approach: Additions of both boron 0.26 at.% and zirconium from 0.3 to 1.5 at.% results in higher strength than exhibited by unalloyed Ni_3Al. The sequence of structural changes of Ni_3Al-based alloy has been correlated with mechanical properties, determined in uniaxial compression tests. Two ranges of work hardening have been identified on the stress-strain curves of these alloys. It was found that the first range of deformation corresponds to the intergranular slip system operating within individual grains, while the second one is connected with transgranular slip.

Findings: Structural observations of A and B alloys showed that zirconium addition causes a decrease of the average grain size. On the mechanical properties of investigated alloys the increasing yield stress and hardness were observed in enlarging zirconium additions. However, the enlarged addition of zirconium causes a decrease of extension. Addition to an intermetallics compound Ni_3Al of such elements as boron (0.26 at.%) and zirconium (0.3 – 1.5 at.%) can be accepted as optimum from susceptibility to plastic deformation point of view.

Research limitations/implications: The main limits for application of the polycrystalline Ni_3Al phases is a poor strength and creep properties at high temperatures. The improvement of mechanical proprieties of Ni_3Al phase required small addition of suitable alloying elements.

Practical implications: The results of investigations as well as the conclusions may be used for improvement of alloys processing based on Ni_3Al intermetallics compound. In well-considered peculiarity selection of alloyed additions and also processing parameters it is possible to steer in a limited range of mechanical properties of these alloys.

Originality/value: The results of investigations expand knowledge about processing of the alloys based on Ni_3Al phase, and in consequence, to apply it in many branches of industry.

Keywords: Ni_3Al intermetallics; Microalloying by B and Zr; Ductilization; Microstructure, Mechanical properties

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MATERIALS

1. Introduction

The Ni_3Al, which has an Ll_2 structure, is an ordered compound which can be a very promising candidate for various high temperature applications. The lack of room temperature ductility and high temperature creep resistance of Ni_3Al in polycrystalline form hinders this compound from practical use.

While a small addition of boron (B) to Ni-rich polycrystalline Ni_3Al resulted in a remarkable improvement in room temperature ductility [l, 2], however, brittleness and low creep resistance at elevated temperature remained as the major barriers for its practical use as a high temperature structural material [3-5].

The many of the studies were to assess the alloy's high-temperature brittleness range and based on that assessment, to determine the alloy's susceptibility to hot cracking [6]. Many of them showed that the tensile and compressive fracture behaviour conformed the strength and ductility variation of the superalloy to temperature. A fracture mechanism involving void formation occurs over a wide range of experimental conditions.

Transgranular and intergranular fracture and cleavage are the other mechanisms operating in a specific temperature range [7-9].

Recent results showed that the substitutional element zirconium (Zr) is an effective element in increasing the creep resistance of Ni_3Al. There were different opinions on the effect of microalloying with Zr on the ductility of Ni_3Al. George et al. [10] reported that Zr has a strong effect on the ductility of Ni_3Al, as long as $Al \leq 24\%$, it is possible to improve ductility by alloying with Zr. In addition, Lia et al.[11] and Chiba et al.[12] found ductilization of Ni_3Al alloys by Zr addition.

However, it was also reported that Ni_3Al alloys doped with a γ'-former (such as zirconium, vanadium, tantalum) exhibited no ductility. Chuang et al.[13] also showed that Zr has no effect on the ductility of B-free polycrystalline Ni_3Al alloys.

In order to know the ductilizing effect and mechanism of Ni_3Al alloys by alloying with Zr, further information is needed. The aim of the present study is to examine the mechanical properties and ductility effects of Zr content of the alloys with the composition of alloy A; Ni 23.7Al 0.26B 0.3Zr and alloy B; Ni 22.5Al 0.26B 1.5Zr (in at. %).

2. Materials and methods

The material was obtained in the form of ingots, which were melted and cast under argon in a vacuum furnace, and then subjected to temperature homogenization 1430 K for 48 h. After initial homogenization treatment, a two phase microstructure with an average primary grain size (of γ') of 40-45 □m was obtained. A second phase (□) comprising about 30 pct by volume fraction, was dispersed as small areas inside the γ' grains. To produce microcracks-free homogenized polycrystalline ingots were isothermally forged by approximately 50% reduction at 1273K and next annealed at 1223K for 24h.

Cylindrical samples for uniaxial compression tests with dimension ϕ 6mm, and h 9 mm high were prepared by spark erosion cutting and next polished mechanically with diamond discs and again annealed at 1223 K for 1h to remove the strain on their surface layer. The uniaxial compression tests were carried out on an Instron testing machine (equipped with protective atmosphere furnace) at room temperature with strain rates $9.25x10^{-5}s^{-1}$,$1.85x10^{-4}s^{-1}$, $1.85x10^{-3}s^{-1}$. The computer data logging of compression force and shortening permitted on the dependence calculation of true stress versus shortening the sample and next on basis of well-known formula the equation of work-hardening curves were determined.

The chemical composition of alloys was carefully analysed with EDS system of Philips XL30 scanning electron microscope after casting and each thermal treatment. As the impurities were below the level of detection by the EDS method, atomic absorption spectrometry method was used. Chemical composition of investigated alloys is shown in Table 1.

Table 1.
Chemical composition of investigated alloys

Alloy	Alloying elements content in at.%				
	Symbol	Ni	Al	B	Zr
Ni_3AlBZr	A	Bal.	23.7	0.26	0.3
	B	Bal.	22.5	0.26	1.5

The alloys were subjected to X-ray qualitative phase analysis conducted on X-ray diffractometer DRON -3. The radiation from Cu-anode lamp with LiF monochromator was used. The length of X-ray wave Cu-lamp, λ_{Cu} was 0,154184nm. Measuring range carried out $20 \div 120^{\circ}$ 2Θ, the exposing time was 6 sec., and the measuring step of angular displacement was 0.02º. The parameters of diffractometer operation: voltage: 42 kV, current intensity: 18 mA. The results of investigations were saved by a computer to complete data. Using Bragg dependence ($2d_{hkl}\sin\Theta = n\lambda$) enumerated the value of lattice plane distance - d_{hkl} .

Experimental values of this distance, as well as the intensity peaks of examined alloys were compared with their board values, where the base of data for identification was used the PDF+4 2006. For structure assessment and potential phase transition of Ni3AlB alloys matrix, before and after zirconium addition, the individual diffraction pattern was compared with the executed samples.

The structure of alloys was studied using an optical microscope - Olympus GX-51 (with possibility of observation in polarized light as well as in Nomarski contrast). The microscope was equipped with a digital system recording images and painting analysis, using special software (DP Soft 3.2). For the purpose of revelations, grains boundary samples were etched in chemical reagent including: 45% CH3COOH, 35% HNO3, 10% HCl, 10% H3PO4 in 5-60 s depending on alloying additions content. Afterwards those the samples were rinsed in ethanol and dried. The remaining images of microstructure were executed on the scanning electron microscope SEM - PHILIPS 525 M with EDAX analyzer (EDS method) equipped with a digital system recording images as well as using Philips XL -30.

3. Results and discussion

The diffraction patterns of alloys: Ni_3AlB as well as Ni_3AlBZr with 0,3 at.% zirconium (alloy A) and 1.5 at.% zirconium (alloy B) are shown in Fig. 1. On these diffraction patterns essential changes of diffractive reflexes position (transition in Ni_3AlB matrix) were not observed.

The series of microstructures studied alloys as cast (a, b) and after homogenization (c, d) are shown in Fig. 2. An alloy after thermal processing (homogenization) is characterized by structure in which the average size of grain is 150 μm smaller than in alloy Ni_3AlB where it is about 200 μm.

Examples of flow curves for alloys A and B determined in compression tests conducted at different strain rate and constant temperature (297 K) are shown in Fig. 3.

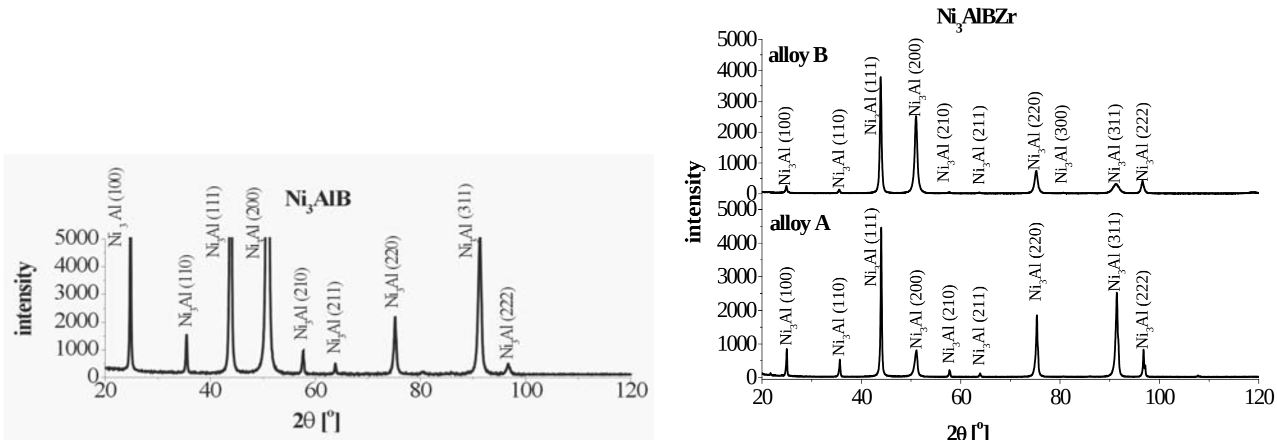

Fig. 1. The diffraction pattern of alloys: Ni_3AlB as well as Ni_3AlBZr, alloy A and B, descriptions on drawings

a) b) c) d)

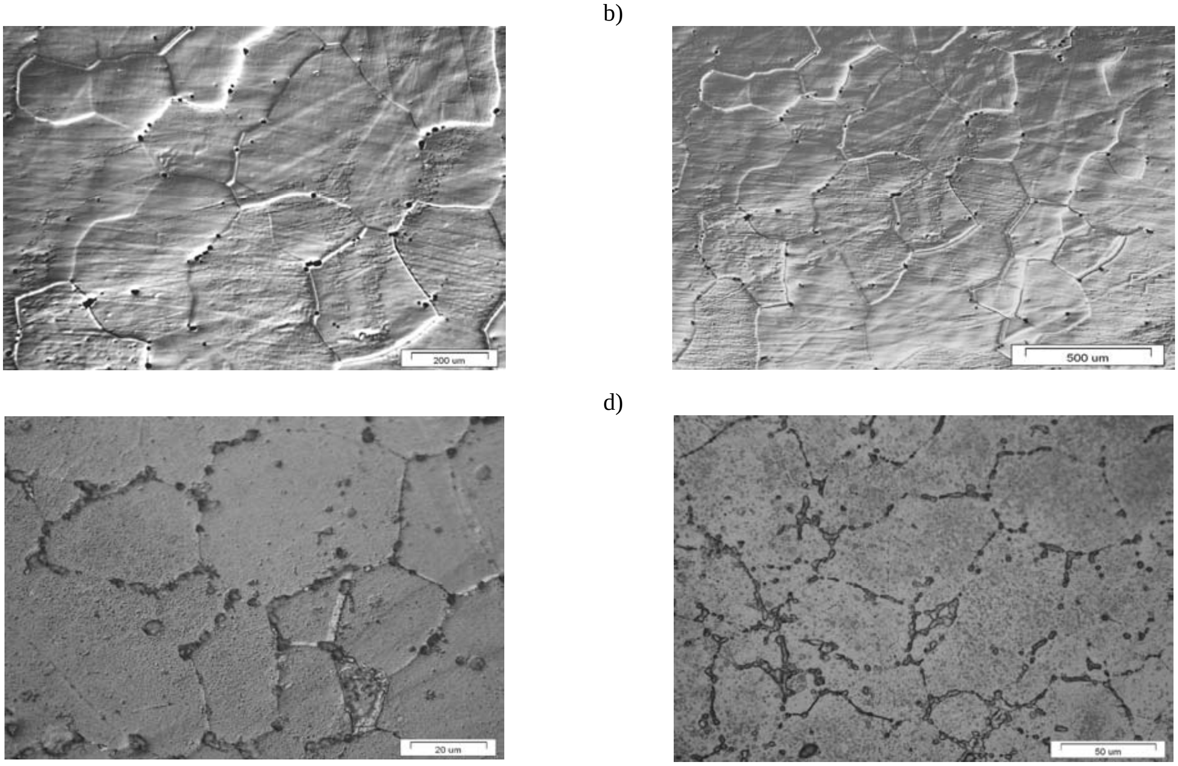

Fig. 2. Microstructure of alloys Ni_3AlBZr (light microscope): a) alloy A, b) alloy B as cast, c) alloy A, d) alloy B after homogenization

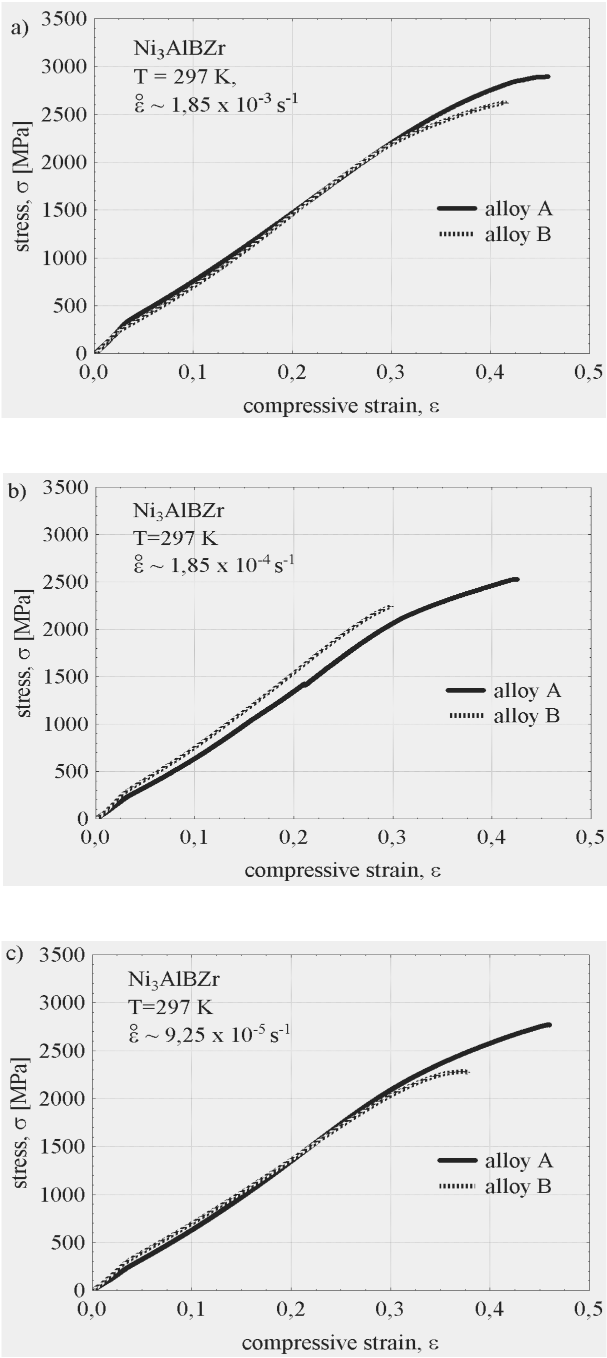

Fig. 3. Flow curves σ = f(ε) for alloys A and B determined in compression tests conducted at strain rate a) 1.85 x10^{-3} s^{-1}, b) 1.85 x10^{-4} s^{-1}, c) 9.,25 x10^{-5} s^{-1} and constant temperature 297 K

It can be observed by the above-mentioned graphs that the larger zirconium addition to the alloy does not influence on strength proprieties significantly, though it characterizes how to be visible with smaller deformability. However, after precise analysing and determining the yield strength R_{02}, as well as hardness HV_{10} (Table 2), growth of the above-mentioned sizes is presented between 1.5 and 0.3 at % zirconium addition.

Table 2.
Hardness HV_{10} and yield strength R_{02} for specimens deformed with different strain rate and a constant temperature

Alloy	Hardness	Strain rate $\dot{\varepsilon}$, s^{-1}		
Ni3AlBZr	HV10	10^{-3}	10^{-4}	10^{-5}
		Yield stress R_{02} , MPa		
A	187 ±0.86%	280	230	224
B	241± 0.82%	330	320	258

Table 3.
Comparison of yield stress (R_{02}) and shortening (A) values for investigated materials

Material	R_{02} , MPa	A, %
Ni_3Al	288	16,6
Ni_3Al + 0, 26%B (at.%)	178	32,8
Alloy A	230	34,3
Alloy B	320	27,4

Comparatively, values of yield stress (R_{02}) and shortening (A) for Ni_3Al, Ni_3Al + 0.26 B (at.%) as well as alloys A and B are put together in Table 3. Compression tests of specimens were carried out in temperature 293 K with strain rate 1.85x$10^{-4}$$s^{-1}$.

It was found that addition 0.3 at.% zirconium to Ni_3AlB alloy resulted in small growth of yield stress and shortening. In the case of alloy B addition 1.5 at. % zirconium causes distinct growth of yield stress, however the shortening undergoes decrease. To analyse the influence of zirconium addition on mechanical properties of investigated alloys graphs, the configuration σ = f (ε, $\dot{\varepsilon}$) has been worked out. Examples of flow curves determined in compression tests conducted at different strain rates and constant temperature 297 K are shown in Fig.4.

The property of the strain hardening in given conditions of temperature and strain rate can be described by a number of functions, among which the most useful for the present purpose is the Hollomon equation $\sigma = K\varepsilon^n$, where K is the strength coefficient and n is the strain hardening exponent [14]. The occurrence of ranges of strain hardening behaviour has been interpreted as the result of deformation mode changes. With regard to the room-temperature deformation of A and B alloys, two strain hardening ranges were observed, having different values of K and n (Fig. 5).

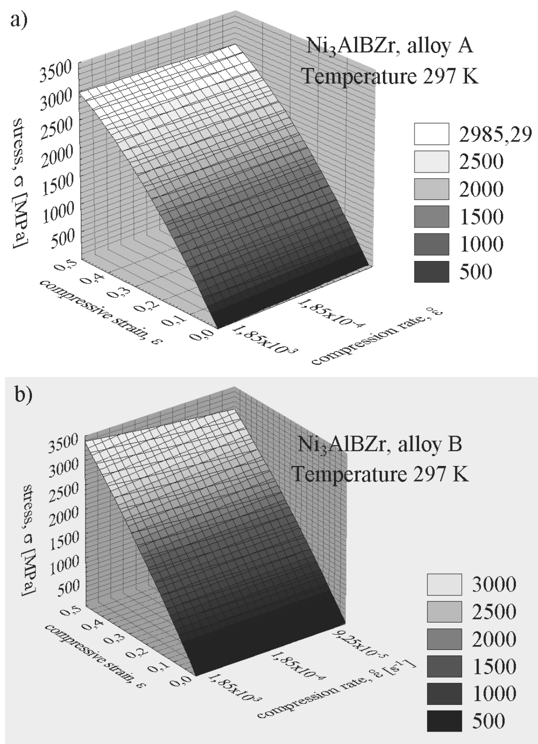

Fig. 4. Mechanical properties of the samples deformations from alloy A and B obtained in the compression tests

The first range was characterized by a higher hardening rate ($n_1>n_2$) and strength coefficient ($K_1>K_2$); the end of this range was associated with a change in the mode of the deformation from the slip within a single grain, to the slip which intersects grain boundaries. The values of strength coefficient (K) and strain hardening exponent (n) for both ranges of strain hardening are summarized in Table 4.

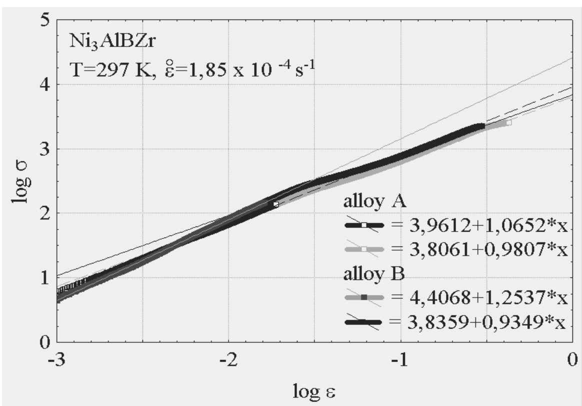

Fig. 5. Strain hardening curves for A and B alloys deformed at a constant temperature and various strain rates

Table 4.
Summary of Hollomon equation coefficients (K and n) and the boundary between two ranges of strain hardening ($\varepsilon_{1/2}$)

Alloy	K_1, MPa	n_1	K_2, MPa	n_2	$\varepsilon_{1/2}$
A	9145.34	1.065	6398.82	0.981	0.0142
B	25852.35	1.254	6456.54	0.935	0.0129

Finding that $K_1 \neq 0$ and $n_1 \neq n_2$, the boundary between two ranges in point on co-ordinates $\sigma_{1/2}$, $\varepsilon_{1/2}$ was marked analytically: $\varepsilon_{1/2} = (K_2/K_1)^{(1/(n1-n2))}$ and $\sigma_{1/2} = K_1(K_2/K_1)^{(n1/(n1-n2))}$

Having marked for every curve work hardening coefficients from Hollomon's equations (K_1, K_2, n_1, n_2) as well as the boundary between ranges ($\varepsilon_{1/2}$) their course may be reproduced in any configuration.

4. Conclusions

In the present study the mechanical properties obtained from compression tests and microstructures of Ni_3AlBZr alloys have been studied as a function of Zr addition (0.3-1.5 at.%), after thermal treatment, deformation at constant temperature and different strain rates.

The following conclusions are drawn:

1. Structural observations of the alloy A; Ni 23.7Al 0.26B 0.3Zr and alloy B; Ni 22.5Al 0.26B 1.5Zr (in at. %) carried out on the light microscope showed that zirconium addition causes decrease of the average grain size in relation to initial state.
2. From the mechanical property of investigated alloys viewpoint increasing yield stress and hardness in enlarging zirconium additions were observed. However, the enlarged addition of zirconium causes decrease of extension.
3. On the basis of the performed investigations it was established that an addition to an intermetallics compound Ni_3Al of such elements as boron (0.26 at.%) and zirconium (0.3 – 1.5 at.%) can be accepted as optimum from the point of view of susceptibility to plastic deformation.

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