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SYNTHESIS AND EVALUATION OF THE STRUCTURE OF Ni₃Al/C ALLOY

1. INTRODUCTION

Metallic matrix composites dispersion reinforced with particles or fibres are considered very interesting structural materials of new generation with wide spectrum of the engineering applications. Some beneficial properties of these materials assumed at an early stage of their design include relatively low density, high melting point, satisfactory electric and thermal conductivity, high resistance to oxidation in the atmosphere of air and resistance to environmental degradation, and as such determine the extended applicability of composites under high-temperature conditions.

Among various composite materials, a very important group form the “in situ” composites, generally considered the engineering materials of a new generation. In the “in situ” composites, the reinforcing phases are generated in a metallurgical process, as a result of various reactions taking place in liquid metal. The materials used most commonly as a matrix of these composites are the following ones: aluminium, magnesium, titanium, cobalt, copper, or their respective alloys. The reinforcing phases are particles of the ceramic materials characterised by high melting point and high hardness like carbides, borides and nitrides of titanium, hafnium, vanadium, tungsten, molybdenum and niobium.

A separate group of the “in situ” composites form the natural composites, also known as anisotropic composites. In these composites, the phases precipitated in eutectic or peritectic reactions are usually formed in the process of directional solidification and, assuming the shape of fibres, lamellae or needles, can play the role of a reinforcement for alloy matrix. Less often they play the role of a lubricating or damping phase, considerably reducing the friction coefficient. This is the role that graphite plays in s.g. cast iron.

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Advanced research works are carried out at present, aiming mainly at the development of a technology for the fabrication of anisotropic „in situ” composites of a new generation, based on the intermetallic Ni_3Al phases with fibrous precipitates of pure carbon, i.e. graphite. The study presented here proves that it is possible to obtain an $\text{Ni}_3\text{Al-C}$ eutectic, not identified so far.

The matrix was formed of an intermetallic Ni_3Al compound with 0.05 wt.% boron addition to improve its plastic properties, while pure graphite was the additional slip phase.

The choice for alloy matrix of an intermetallic phase from the aluminium – nickel system was dictated, among others, by the possibility of making this phase more plastic with an addition of boron, by a high value of the tenacity (R_m/γ), high resistance to oxidation in a wide range of temperatures, and also by the mechanical properties increasing with temperature increase up to 923–1123 K. Other characteristic features of this compound include its high creep resistance and resistance to abrasion wear, especially at high temperatures, combined with resistance to cavitation erosion.

Owing to their beneficial technological and mechanical properties, the intermetallic phases, particularly aluminides from the Ni-Al group, are nowadays gaining importance as a very attractive structural material. Yet, because users of these products vary in their requirements, new materials of always better functional properties are intensely searched for.

2. EXPERIMENTAL

2.1. Synthesis of $\text{Ni}_3\text{Al/C}$ alloy

The synthesis of Ni-Al alloys is often accompanied by an exothermic reaction (exomelt), which determines the run of a metallurgical process. A very important element of the cost-effective melting process is the way in which the charge materials are stacked in a crucible. A schematic representation is shown in Figure 1.

For the synthesis of alloy based on an intermetallic $\text{Ni}_3\text{Al}+\text{C}$ phase, a BALZERS vacuum furnace was used. Melting was carried out in the atmosphere of argon, at a constant power feed of 5 kW, and at a temperature of about 1600°C. The weighed batches of aluminium and nickel were selected in such a way as to produce later a phase of stoichiometric composition. Aluminium is the first one to melt. Nickel dissolves in aluminium at a temperature of about 800°C, and the process is of strongly exothermic nature. Due to this, the temperature raises considerably and melting of the remaining nickel batch is proceeding at a very high rate. The exothermic reaction between aluminium and nickel takes the time of a few seconds only, but owing to sudden and rapid increase of temperature, considerable savings in the electric energy consumption are possible. At a temperature of about 1600°C, fine pieces of the spectrally pure carbon were introduced to Ni-Al melt. With the last batch of carbon, pure cerium was also added (cerium is used as a spheroidising agent in common iron-carbon systems). After solidification of the Ni-Al alloy, an Ni_3Al phase containing graphite in its structure was formed. The shape of graphite precipitates was determined by physico-chemical investigations. The obtained material was next poured into a ceramic “Y” shaped mould, which before pouring with liquid metal was preheated to a temperature of about 500°C.

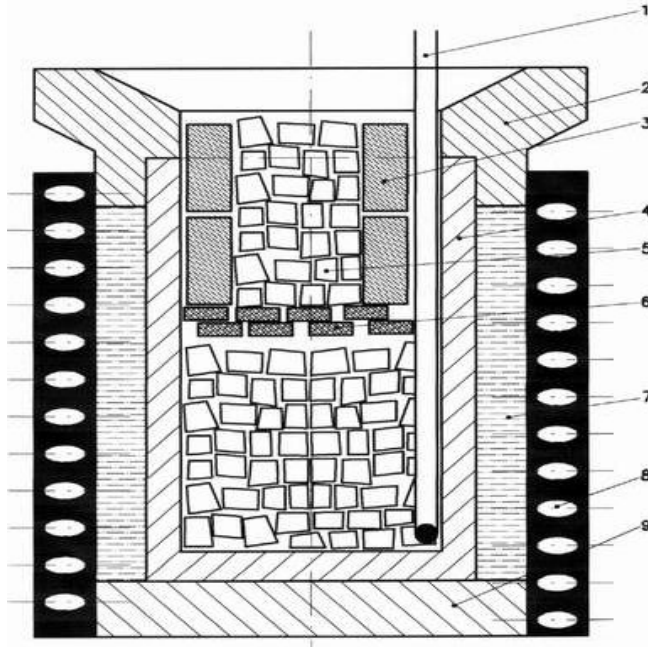


Fig. 1. Schematic representation of charge position in crucible (Exo-Melt ProcessTM [6]), 1 – Pt-Pt 18Rh thermocouple; 2 – graphite top coated with Morgan cement; 3 – aluminium; 4 – ceramic crucible (Al_2O_3); 5 – nickel; 6 – alloying elements (Al-B master alloy); 7 – spinel powder (Al_2O_3 , MgO, ZrO₂); 8 – induction coil; 9 – ceramic element (Al_2O_3)

2.2. Method of investigation

From the “Y” wedge-shaped casting, two specimens designated as A and B were cut out for the metallographic examinations (Fig. 2).

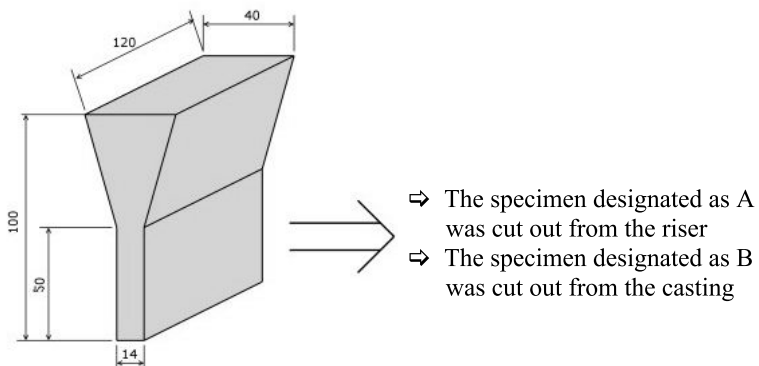


Fig. 2. Schematic representation of “Y” shaped casting from which metallographic specimens were cut out

The phases (regions A and B) were examined under a metallographic microscope and a JEOL JSM-5500 LV scanning microscope. The scanned images of the specimen surface were recorded in an Se mode (topography) and Comp mode (qualitative chemical composition). In parallel, the X-ray spectrum was analysed, determining in microregions the chemical composition of precipitates by an ED X-ray fluorescence method.

2.3. Results and discussion

Figures 3 to 4 show the microstructure of specimens in region A and in region B, respectively.

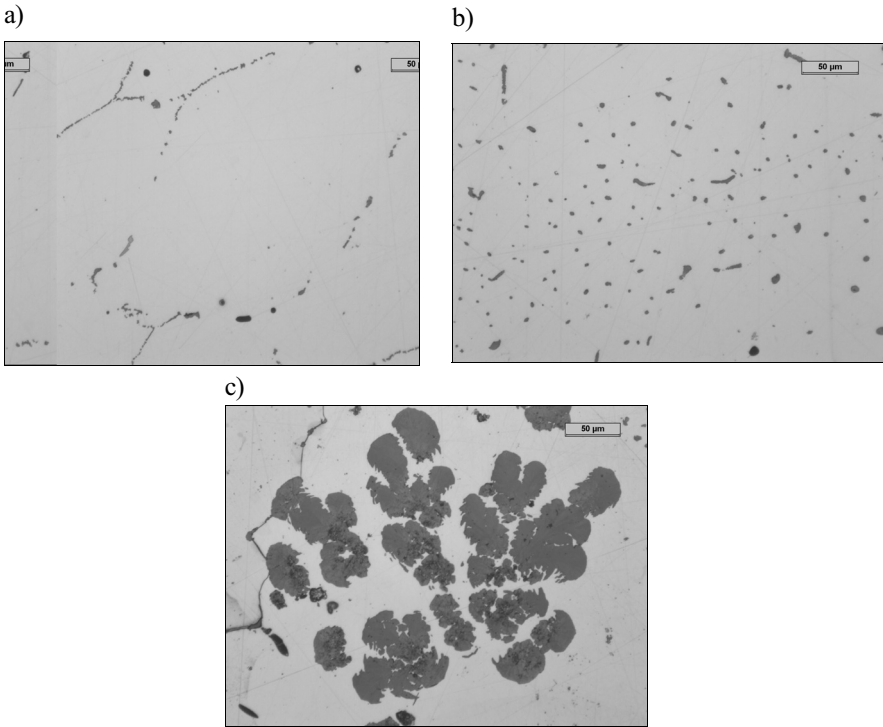


Fig. 3. Microstructure of region A, 250×: a) the well-visible light area is Ni_3Al phase forming alloy matrix, the fine dark precipitates appearing along the grain boundaries are pure carbon in the form of graphite precipitates; b) observe numerous dark spherical carbon precipitates and traces of fibrous graphite eutectic; the light area is Ni_3Al phase; c) the well-visible light area is Ni_3Al phase forming alloy matrix, the large dark precipitates of 50 μm dimensions which appear in central part of the image are pure carbon in the form of graphite

Etching of specimens revealed the presence of two phases. The light region forming alloy matrix was the Ni_3Al phase. The dark phase showed different morphologies, since it crystallised in three different forms:

- **First form:** fine precipitates of oblong shapes appearing at the grain boundaries,
- **Second form:** spherical precipitates, which most probably mark the place of intersection with normal fibre planes, similar to those that are formed in a eutectic cell,
- **Third form:** large precipitates of a fuzzy spherical phase resembling temper carbon in cast iron.

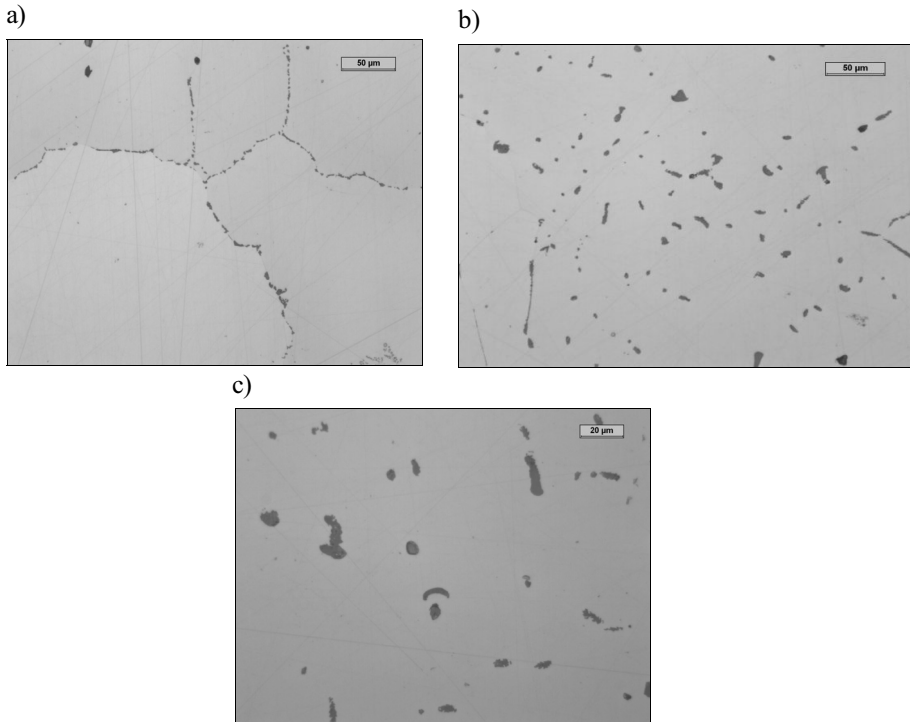


Fig. 4. Microstructure of region B: a) 250×, etching revealed the grain boundaries along which numerous and very fine precipitates of pure carbon are visible; the light area is Ni₃Al phase; b) 250×, the dark areas of a circular shape are carbon precipitates, while the light area is Ni₃Al phase forming alloy matrix; c) 500×, observe the dark precipitates of pure carbon characterised by irregular and oblong shapes; the Ni₃Al phase visible as a light area is alloy matrix

Similar observations were carried out on the specimen designated as B. The two-phase structure is well visible; the light region is alloy matrix, while the dark phase is composed of graphite precipitates in different forms.

In specimens from region B, the spherical forms of graphite existed no longer. Further examinations were carried out by SEM. Figures 5 to 12 show the results of Se analysis (topography) of specimens from regions A and B. The specimens show the presence of numerous precipitates of a dark phase (carbon) and light phase (Ni₃Al), which is alloy matrix. The examinations have confirmed the results obtained during metallographic examinations.

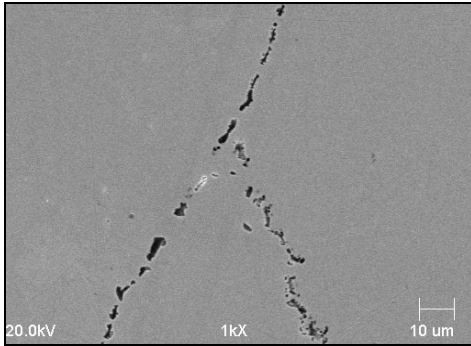


Fig. 5. Specimen A, 1000 \times . The dark region is composed of graphite precipitates which appear along the grain boundaries; the light region is Ni_3Al phase forming alloy matrix

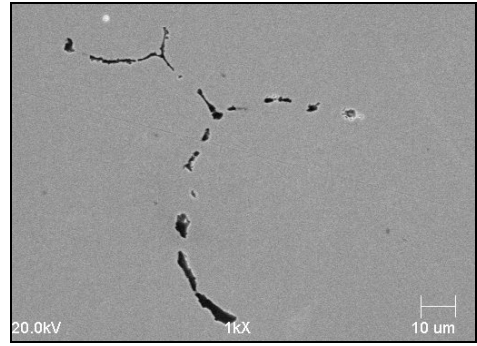


Fig. 6. Specimen B, 1000 \times . Visible are numerous dark graphite precipitates which appear along the grain boundaries; the light region is Ni_3Al phase forming alloy matrix

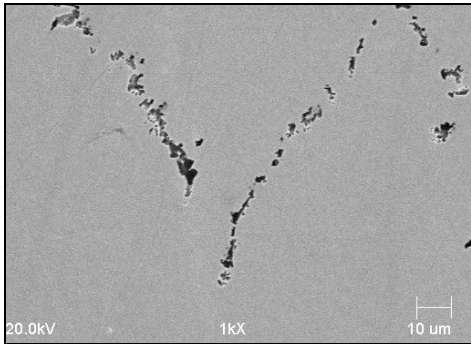


Fig. 7. Specimen A, 1000 \times . Visible are numerous irregular dark carbon precipitates, the light region is Ni_3Al phase

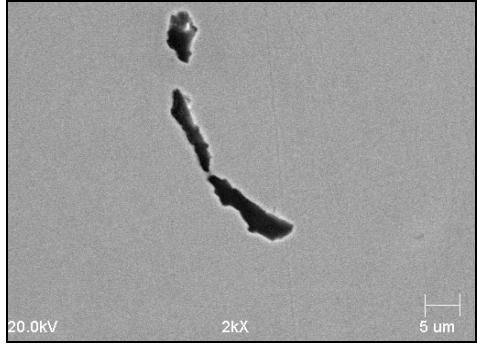


Fig. 8. Specimen B, 2000 \times . The light region is an intermetallic Ni_3Al phase forming alloy matrix, while the dark region is carbon

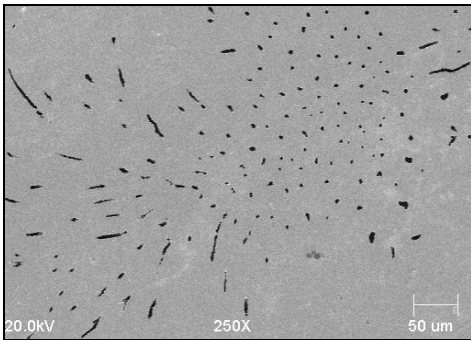


Fig. 9. Specimen A, 250 \times . Visible are numerous dark circular and acircular precipitates of eutectic graphite, the alloy matrix is light Ni_3Al phase

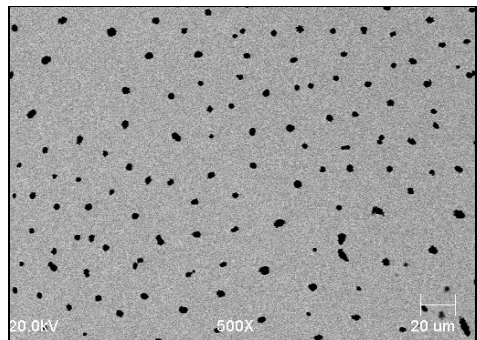


Fig. 10. Specimen B, 500 \times . Visible are numerous fine and regular carbon precipitates of a dark colour, the light region is an intermetallic Ni_3Al phase

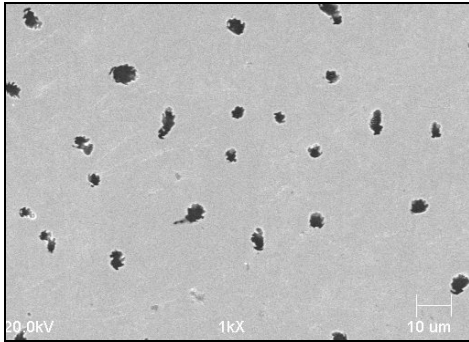


Fig. 11. Specimen A, 1000×. Observe spherical precipitates similar to those that are formed in a eutectic cell of cast iron, the alloy matrix is light phase, i.e. Ni_3Al

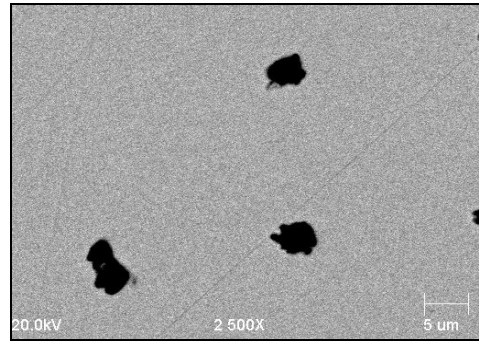


Fig. 12. Specimen B, 2500×. Visible at large magnification are the precipitates of pure carbon in the form of spheres, the alloy matrix is Ni_3Al phase visible as a light region

To identify certain phases and determine the chemical composition of alloy matrix, an EDX-ray analysis was carried out in selected microregions.

It has been stated that in specimens A and B the matrix consisted of an aluminium-nickel alloy of the following composition:

- aluminium 10.5 wt.%,
- nickel 89.5 wt.%,

which after solidification formed a homogeneous nickel aluminide phase of an Ni_3Al type.

3. CONCLUSIONS

- Preserving constant operating parameters of a Balzers vacuum furnace, the Al-Ni alloy has been produced by EXOMELT technique, ensuring a cost-effective run of the melting process.
- The said alloy after solidification forms an Ni_3Al phase, homogeneous in respect of its physico-chemical properties.
- The Ni-Al-C alloy was fabricated by a common process. The process of carbon transfer to solution under argon protective atmosphere and at a negative pressure of 0.05 MPa took the time of about 300 seconds.
- The microstructure of Ni_3Al -C alloy specimens revealed different forms of graphite morphology, from the precipitates on grain boundaries, through fibrous forms up to the graphite in the form of spheroids.
- As regards the morphology of carbon precipitates in the examined alloy, one can observe a similarity to Fe-C alloys with high carbon content.

Acknowledgements

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