Effect of carbon on structural changes in Ni$_3$Al phase

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Abstract

The paper presents diffraction and microscopic researches of a carbon influence on structural changes in Ni$_3$Al phase. Ni$_3$Al phase was acquired thanks to vacuum casting technology with a use of Exo-Melt™ process. A casting process was conducted in the atmosphere of argon. Two basic alloys were made with maintaining the nominal nuclear share equivalent to stoichiometry of Ni$_3$Al phase. Next, carbon was instilled into the liquid alloy in the rate of 0.2% and 1.25% respectively for the first and for the second alloy. Final casts were cut into pieces and the samples were prepared in order to undertake and microscopic researches. Their phase composition was analyzed as well as their structural changes occurring within Ni$_3$Al phase. Some changes along the lattice constants of Ni$_3$Al phase were noticed, and they were 0.3580 nm and 0.3589 nm respectively for 0.2% C and 1.25% C. In the alloy consisting of 2.25% C a graphite (001) atomic plane was noticed. Its existence is connected with transgression in solubility limit of carbon in Ni$_3$Al phase. This result was also confirmed by researches with SEM imaging, in which we observed numerous of separation of graphite of lamellar and nodular shape.

Keywords: Intermetallics, Nickel aluminides, Ni$_3$Al, X-Ray

1. Introduction

Intermetallic phases, thanks to their properties such as high melting point, low density, high durability, and resistance to corrosion and oxidation in high temperature conditions, constitute an interesting group of construction materials. Recently they have been used only in components which work under increased temperature due to their fragility and low ductility in ambient temperature [1].

An example of an intermetallic phase with interesting durability parameters is Ni$_3$Al. In this phase we can observe a reverse tendency comparing to most of conventional alloys and it is connected to an abnormal increase of durability parameters in a function of temperature [2].

Durability properties of Ni$_3$Al phase can be increased by applying hard and heat-resistant ceramic phases of carbides and borides types [3]. One of the way enabling this process is the SHSB method (Self Propagating High-Temperature Synthesis in Bath) [4,5]. It consists of adding to liquid alloy compressed packets of stoichiometric mixture of metal-nonmetal powders, providing SHS reaction.

The main method of getting Ni$_3$Al phase, similar for other nickel aluminides, is the Exo-Melt™ process [6], the thermal explosion method and hot extrusion (TE/HE) [7] as well as the method of SHS/HE powders synthesis [8].

Structurally, Ni$_3$Al phase is classified to the space group Pm3m with aluminum nuclei in nodes and nickel on its walls [9]. A lattice constants that phase is 0.357 nm. Different nuclear diameters nickel and aluminum guarantee acceptable volume of tetra and octahedral gaps, in which one may allocate nuclei of other elements such as carbon or boron [10].

In the presented paper we presented researches about structural changes which take place in Ni$_3$Al phase under an influence of added carbon. The aim of that paper was defining...
changes of nest structural parameters of Ni₃Al phase and examining tensions in alloys consisting respectively 0,25% and 1,25% C. The crucial point of the examination was a detection of other structural components formed as a result of exceeding the maximum solubility of added carbon alloys.

2. Experimental procedure

A chemical composition of alloys used for researches is presented in Table 1. The basic chemical composition used for all melts was corresponded to stoichiometry of Ni₃Al phase.

Table 1. Chemical composition of alloys

<table>
<thead>
<tr>
<th>Melt No.</th>
<th>Ni [at.%]</th>
<th>Al [at.%]</th>
<th>C [wt.%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>75</td>
<td>25</td>
<td>0,2</td>
</tr>
<tr>
<td>2</td>
<td>75</td>
<td>25</td>
<td>1,25</td>
</tr>
</tbody>
</table>

Nickel and aluminium required for Exo-Melt™ synthesis of Ni₃Al phase, was prepared according to nominal atomic ratio of Ni/Al = 3:1. In order to achieve it, nickel and aluminium was used with purity 99,98% and 99,7% respectively. Graphite with spectral purity 99,999% was added in form of pressed rods of diameter 0,005 m and length 0,05 m, calculating its weight share according to the mass charge corresponding to stoichiometry of Ni₃Al phase.

The melt mould were made of molochite and sodium-water glass in proportions 10:1 and next they were hardened thanks to CO₂ blowing. A scheme of the mould is presented in Fig. 1. Afterwards mould were dried in temperature 773 K for 10 minutes and then they were placed one by one in a chamber of vacuum furnace. A melt was conducted in initial vacuum conditions 5x10⁻⁴ MPa and it was flooded in atmosphere of pure argon in pressure of 5x10⁻² MPa. Weight of basic alloy input for all melts was 0,5 kg.

The microstructural characterization of all alloys were carried out on scanning microscopy (SEM) and optical microscope (OM). The chemical composition in micro-area was examined with X-ray microanalysis (EDX). The phase analysis was conducted with a use of X-ray diffraction (XRD) with a Cu Kα radiation at 45 kV and current 40 mA. Samples for the XRD research, of size 10x10x10 mm, were taken from the ingot core. A quality analysis of materials was done in Bragg-Brentano geometry on polycrystalline material. The stress in the materials were examined directly ion samples taken from casts according to sin² method (Psi) Chi-Phi-x-y-z Cradle. The scanning parameters are presented in Table 2.

Table 2. The scan parameters used for the stress measurements

<table>
<thead>
<tr>
<th>Step size (°2θ)</th>
<th>Range (°2θ)</th>
<th>Step size (Sin²(ψ))</th>
<th>Range (Sin²(ψ))</th>
<th>Counting time (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0,2</td>
<td>86,1-93,9</td>
<td>0,1</td>
<td>0-0,9</td>
<td>10</td>
</tr>
</tbody>
</table>

3. Results

Figure 2 presents results of phase analysis of cast alloys No. 1 and No. 2, which components are presented in Table 1. The two examined alloys consist Ni₃Al phase. Alloy No. 2 consists additional structural component, which 002 peak in 25,6º 2Θ corresponds to graphite surface (001). The increase of 1,25% addition of carbon in alloy No. 2 influenced on the change of diffraction image. Two additional peaks appear 100 and 110 of Ni₃Al phase. These reflections were not noticed with alloy diffractogram with lower amount of carbon.

X-ray analysis of alloys No. 1 and No. 2 is presented in Figure 3 with visible 111 peaks shift from the (111) atomic plane Ni₃Al.
phase. The peak position is 43,785° and 43,575° respectively for samples with 0.2% C and 1.25% C.

![Graph](image1.png)

Fig. 3. Comparative X-ray diffraction patterns of 111 peak shift from the (111) atomic plane Ni$_3$Al phase

According to diffractograms of both studied materials, we defined parameter of regular net of Ni$_3$Al phase, and we have taken into consideration Nelson-Riley’s extrapolation function in calculations. It was 0.3580 nm and 0.3589 nm respectively from alloys consisting 0.2% and 1.25% of carbon.

Thanks to using scanning parameters presented in Table 2 we conducted researches of internal tensions in the structure of Ni$_3$Al phase for (311) atomic plane. Whey are 510 MPa and 12 MPa respectively from samples with 0.2% and 1.25% of carbon.

On Figure 4 of alloy consisting 0.2% of carbon is presented. In this material we can see single phase microstructure, consisting of Ni$_3$Al phase grain.

![Image](image2.png)

Fig. 4. An image of SE Ni$_3$Al alloy consisting of 0.2% of carbon. Mag. 500x

The microstructure consisting of 1.25% of carbon which is visible on Figure 5 is two phase and it consists of structure (light color) which is Ni$_3$Al phase and it also consists of graphite separations (dark color).

![Image](image3.png)

Fig. 5. The microstructure of alloy consisting of 1.25% of carbon with visible graphite morphology in a matrix of Ni$_3$Al phase. The image obtained thanks to optical microscopy

A morphology of the graphite in the microstructure is varied. One can observe either few nodular (Fig. 6) or lamellar precipitation (Fig. 7).

![Image](image4.png)

Fig. 6. SEM-SE image precipitates of nodular graphite in Ni$_3$Al matrix. Deep etching with aqua regia
3. Discussion

The conducted diffraction study confirmed structural changes in Ni₃Al phase caused by carbon addition to the alloy. The comparison of X-ray diffraction patterns of both examined materials, consisting of 0.2% and 1.25% of carbon reveal also some phase changes. They deal mainly with an appearance of peak in alloy with increased carbon presence in 25.6° 2θ characteristic for graphite (001) atomic plane (Fig. 2). Its presence is connected with exceeding the solubility limit of carbon in Ni₃Al phase and with the production of excess carbon nuclei in form of graphite (Figs. 5-7). The effect is confirmed by calculating the lattice constants of Ni₃Al phase, which was 0.3580 nm for the alloy consisting of 0.2% of carbon, whereas in the alloy with 1.255 its size increased by 0.3589 nm. A tendency to change volume of regular crystal structure of Ni₃Al phase evidences that octahedral and tetrahedral spaces are filled with carbon nuclei. In Ni₃Al phase the size of octahedral and tetrahedral spaces is respectively 55 pm and 30 pm.

The lattice parameter of Ni₃Al phase was established based on received diffractograms and with using the Nelson-Riley extrapolation function.

4. Conclusions

The conducted X-ray diffraction studies have shown changes in the structure and phase components in alloys No.1 and No.2 according to the addition of carbon.

With the addition of carbon on the level of 1.25 in alloy structure No.2, graphite appears.

The carbon added to liquid alloy, according to stoichiometric of Ni₃Al phase, influences the increase of its lattice constants.

The exceeding of solubility limit of carbon in Ni₃Al phase, influences the production of graphite in lamella and nodular form.

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References