Abstract
Creep characteristics were determined in directionally solidified Ni$_3$Al based alloy. Foundry alloy was alloyed by zirconium and its nominal composition was Ni-22Al-1Zr. Foundry alloy was prepared by method of vacuum induction casting, it was melted in a corundum crucible and cast into a graphite mould. The rods manufactured in this way were directionally solidified by Bridgman's method in corundum tubes with a specific angle. Rate of directional solidification was 20 and 50 mm/h. Test specimens with length of 62 mm and body diameter of 3.5 mm were turned out from central parts. The specimens thus prepared were suitable for determination of basic high temperature characteristics. The following test parameters were chosen for uninterrupted creep tests – testing temperature of 900°C and applied nominal load of 80 MPa. The determined creep characteristics differ significantly in dependence on conditions of directional solidification. Very distinct differences were found in time to rupture and in elongation. Determined rates of secondary creep are, however, similar and their value is approx. 8.3·10^{-8} s$^{-1}$. This value is satisfactory for this type of material. Material structure before and after creep was investigated on cross sections; phase composition, micro-hardness and material porosity were determined.

Keywords: Ni$_3$Al based intermetallic compounds, creep test, creep rate, structure

1. INTRODUCTION

Issues related to high-temperature creep of Ni$_3$Al based alloys are subject of a long-term interest [1, 2]. These alloys can be used, thanks to their favourable properties, for high-temperature applications also in corrosive environment. Single phase alloys Ni$_3$Al [3] rose special interest, as they offered favourable properties especially in monocristalline state. Preparation of single crystals of this type of alloy is highly demanding. That’s why poly-crystalline alloys were tested more frequently. Single phase unalloyed alloys do not achieve such good results as alloyed multi-phase alloys [4]. Structure of unalloyed and alloyed alloys can be very well influenced by process of directional solidification, which makes it possible to grow uni-directed multi-phase alloys, which can be considered as metal matrix composites.
2. EXPERIMENTAL PART

Foundry alloy was alloyed by zirconium and its nominal composition was Ni-22Al-1Zr. Foundry alloy was prepared by method of vacuum induction casting, it was melted in a corundum crucible and cast into a graphite mould. The rods manufactured in this way were directionally solidified (DS) by Bridgman’s method in corundum tubes with a specific angle. Directional crystallisation was realised in a two-zone crystallisation furnace. Rate of directional solidification \( r_{DS} \) was 20 and 50 mm/h. Figure 1 shows a rod after directional solidification. The samples prepared in this manner were then used for determination of structural characteristics and for manufacture of testing rods.

![Fig.1 Rod made of Ni-Al-Zr alloy after directional solidification](image)

2.1 Evaluation of mechanical characteristics

Creep characteristics were determined in directionally solidified Ni$_3$Al based alloy. Test specimens with length of 62 mm and body diameter of 3.5 mm were turned out from central parts. The specimens thus prepared were suitable for determination of basic high temperature characteristics. The following test parameters were chosen for uninterrupted creep tests – testing temperature of 900°C and applied nominal load of 80 MPa. The following table summarises determined values of high-temperature characteristics of inter-metallic compounds Ni-Al-Zr.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Content [at.%]</th>
<th>( r_{DS} ) [mm/h]</th>
<th>Time to rupture [h]</th>
<th>Elongation A [%]</th>
<th>Reduction Z [%]</th>
<th>Rates of secondary creep [s$^{-1}$]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Ni-22Al-1Zr</td>
<td>50</td>
<td>1190</td>
<td>34.6</td>
<td>54.4</td>
<td>8.3$\cdot 10^{-8}$</td>
</tr>
<tr>
<td>2</td>
<td>Ni-22Al-1Zr</td>
<td>20</td>
<td>690</td>
<td>20.2</td>
<td>29.0</td>
<td>8.3$\cdot 10^{-8}$</td>
</tr>
</tbody>
</table>

The above results of high-temperature characteristics were determined on the basis of correctly performed tests. Test specimens were fractured in the measured area and testing temperature varied during testing within an interval of max. ± 1°C. The value of determined elongation A corresponds very well with the results of continuous measurement of deformation. The determined creep characteristics differ significantly in dependence on conditions of directional solidification. Very distinct differences were found in time to rupture and in elongation. Determined rates of secondary creep are, however, similar and their value is approx. 8.3$\cdot 10^{-8}$ s$^{-1}$. This value is satisfactory for this type of material. Figure 2 shows dependencies of relative deformation and time for the samples 1 and 2. Slight indirectness in diagrams is caused by an extreme sensitivity of the measurement device, it is not characteristic manifestation of the material response.
2.2 Evaluation of structural characteristics

Material structure before and after creep was investigated on cross sections. Structure of the sample was observed by optical and scanning microscope. The samples were multi-phase. Figures 3 and 5 show structures of the samples 1 and 2 after directional crystallisation. Figures 4 and 6 show structures of the samples 1 and 2 after creep. Phase Ni$_3$Al occurs here with aluminium content ranging from 24 to 22 at.%, as well as phase with lower aluminium content corresponding to nickel (Ni) solid solution, and phase rich in zirconium. Phase containing approx. 22 at.% of aluminium contains approx. 3 at.% of Zr. It was established after evaluation and comparison of the determined composition of the phase rich in Zr with zirconium content of approx. 17.5 at.% with ternary diagram [5], that most probably this was the phase Ni$_5$Zr, which may occur within the interval from 14.85 to 18.4 at.% Zr.

Fig. 2 Creep diagram of the samples 1 and 2

Fig. 3 Sample No. 1, microstructure after DS

Fig. 4 Sample No. 1, microstructure after creep
Fig. 5 Sample No. 2, microstructure after DS

Fig. 6 Sample No. 2, microstructure after creep

Fig. 7 Sample No. 1, microstructure after DS (BEC)

Fig. 8 Sample No. 1, microstructure after creep (BEC)

Fig. 9 Sample No. 2, phase analysis (BEC)
Figure 7 shows structure of the sample 1 after directional solidification observed by scanning microscope. Figure 8 shows structure of the sample 1 after creep in deformed area. Light particles represent the phase rich in Zr. Dark areas are holes formed after mechanical load. Identical phase are present in the samples after directional solidification and after creep. Figure 10 specifies individual identified phases on cross-section of the sample 2. It was impossible to make more precise determination of the area with the phase rich in Zr. Micro-hardness was determined on cross-sections. Its value does not differ significantly in uni-directed state and after creep, and it is approximately within the range of 309-335 HV0.05. Porosity was determined on cross-sections by image analysis. The value of porosity on the sample 1 in uni-directed state and after creep is within the range of 0.04-0.05 %, it is slightly lower in case of the sample 2 – namely 0.02-0.03 %.

3. CONCLUSIONS
Creep characteristics were determined in directionally solidified Ni$_3$Al based alloy. The specimens thus prepared were suitable for determination of basic high temperature characteristics. The following test parameters were chosen for uninterrupted creep tests – testing temperature of 900°C and applied nominal load of 80 MPa. The determined creep characteristics differ significantly in dependence on conditions of directional solidification. Very distinct differences were found in time to rupture and in elongation. Determined rates of secondary creep are, however, similar and their value is approx. 8.3·10$^{-8}$ s$^{-1}$. This value is satisfactory for this type of material. Material structure before and after creep was investigated on cross sections. Identical phases are present in the samples after directional crystallisation and after creep. They differ only by their size and placing.

LITERATURE


ACKNOWLEDGEMENT

The presented results were obtained within the frame of solution of the research project MSM 6198910013 „Processes of preparation and properties of high-purity and structurally defined special materials“. We thank Ing. K. Konečná from VŠB-TUO for realisation of analyses of phase composition.