Kinetic of the Intermetallic Phases Growth in the Al Alloy - Ni Composite

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This article describes the kinetic of intermetallic phases growth during annealing of Al alloy – Ni composite material. The composite was prepared by spark plasma sintering processing (SPS) from gas atomized AlCr₆Fe₂Si₁ with 5 wt. % of Ni. The composite material is stable at temperatures up to 400 °C and no formation of intermetallic phases was observed even after 100 h of annealing. At higher temperatures, the intermetallic phases are formed and the time of the first observation of the intermetallics is strongly dependent on temperature. The only intermetallic phase that was proven by the XRD was the NiAl₃. The content of other intermetallic phases, that were clearly visible on the SEM micrographs, was probably lower than the detection limit of the XRD. The forming layers of intermetallics served not as a protective barrier and the reactions continued until the whole amount of Ni reacted, as was established by observation of the sample annealed for 100 h at 550 °C.

Keywords: Al alloys, Intermetallics, SPS, self-healing

1 Introduction

Rapidly solidified aluminium alloys are promising materials with increased strength and resistance to elevated temperatures compared to the common aluminium alloys. It was proven that AlCr₆Fe₂Si₁ alloy prepared by gas atomization can be successfully compacted by hot extrusion [1], spark plasma sintering [2] and high-pressure spark plasma sintering [3]. All these compact materials exhibit good mechanical properties and thermal stability up to 450 °C [1].

The formation of intermetallic phases in the Al-Ni system is effortless and can be performed even in the air atmosphere [4]. This fact provides a background to one of the possible self-healing mechanisms in the aluminium alloys which mechanism is illustrated in Fig. 1.

In the Al alloy – Ni composite material, the intermetallic phases are growing under the temperature of 550 °C. The intermetallic phases are growing in the concentric layers around Ni core with different stoichiometry. The phase which is in touch with Al matrix is the NiAl₃ and grows in radial dendrites. These dendrites are closing cracks and voids in the material and are responsible for the self healing behaviour [5].

The topic of this paper is to study the kinetic aspect of this process. The information about the precise time of intermetallic formation will help to optimize the heat treatment process for closing of the cracks.

2 Experimental

The alloy with composition of Al- 6 wt. % Cr-2 wt. % Fe-1 wt. % Si was prepared by melting appropriate
amount of pure metals. Subsequently the alloy was processed by gas atomization. The powder was mixed with 5 wt. % of gas atomized Ni and the mixture was sintered by SPS (FCT HP D10) at 500 °C with duration of 15 min. Bulk samples were observed by SEM (TESCAN VEGA 3 LMU) and phase composition was measured by XRD (PANalytical X’Pert PRO, Co lamp). The annealing was performed at 400, 450, 500 and 550 °C and it lasted for 0.1, 0.25, 1, 10 and 100 h for each temperature.

3 Results and discussion

The initial material was formed by Al matrix, Al₁₃Cr₂ and Al₁₃Fe₄ intermetallic phases and Ni particles, as shown in Fig. 2. The conditions of the SPS compaction did not lead to the formation of any intermetallic phase on the boundary between Al based matrix and Ni particles.

Fig. 2 Microstructure of the initial material (SEM)

Fig. 3 Microstructure of materials after first intermetallic phases occurrence after annealing at different temperatures
The time necessary for the occurrence of intermetallic phases was different for each temperature. At the lowest temperature (400 °C), the phases were not observed even after 100 h of annealing, see Fig. 3. This proves excellent thermal stability of the material. It also shows that the material could be used up to this temperature without unwanted degradation.

The first occurrence of the intermetallic phase was observed after 10 h during annealing at 450 °C. This fact has two consequences: The material is not suitable for long-term exposition at this temperature due to the intermetallic phases formation. On the other hand, the time to intermetallic phase formation is too long to be applicable for self healing of the material. During the annealing at 500 °C, the very first intermetallic phases were observed after 1 h. Although, the time for the intermetallic phases formation is shorter, the outcomes are the same as described earlier for the temperature of 450 °C. The situation is different for the temperature of 550 °C. The first intermetallic particles were observed after 0.1 h (6 min.) of annealing.

As shown in Fig. 4, the growth of intermetallic phases continued with time of annealing. After 0.25 h, the layer of intermetallic phases was still not sufficient. After annealing for 1 h, the complex shell structure was observed. On the boundary of the residual Ni particle, the NiAl phase is located. The following compact phase is Ni$_2$Al$_3$ and the phase formed by dendrites growing in the Al matrix is the NiAl$_3$. The phase composition was evaluated based on measuring of local chemical composition by the EDS. The XRD proved only presence of NiAl$_3$, as shown in Fig. 5. The content of other phases is probably under the detection limit. The increase of annealing time from 1 h to 10 h only increased the amount of NiAl$_3$. After 100 h of annealing, the Ni completely reacted forming NiAl$_3$ phase. The material approached the equilibrial state. According to Al-Ni phase diagram [6], the alloy with 5 wt. % of Ni should be formed of eutectic of Al and NiAl$_3$. 

Fig. 4 Microstructure of material annealed at 550 °C with different duration
Although the intermetallic phases were observed after 0.1 h of annealing, they were detected by XRD in samples annealed for 1 h and longer, see Fig. 5.

4 Conclusion

In this paper was proven that intermetallic phases in the Al alloy-Ni system are formed in solid state. The prepared composite material is stable up to 400 °C. The formation of intermetallic phases was observed starting from the temperature 450 °C. The only detected intermetallic phase was NiAl₃, although presence of small amounts of phases with different stoichiometry is highly probable. After sufficient time of annealing depending on temperature, the Ni reacted completely.

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