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Microstructure Evolution of Directionally Solidified Al-12%Ni Hypereutectic Alloy (Post-print)

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Abstract

Using the Bridgman directional solidification technique, constant-velocity directional solidification and transition-accelerated directional solidification experiments were performed on Al-12%Ni (mass fraction) hypereutectic alloy within a growth rate range of 1~100 m/s under various initial growth conditions and velocity ratios to determine the conditions for obtaining coupled eutectic structures. The results revealed that at a growth rate of 1 m/s, after solidifying over a certain distance, the microstructure exhibited no independently growing primary phase, yielding a coupled eutectic structure; however, at growth rates of 2~100 m/s, the primary Al_3Ni phase grew preferentially, displaying typical faceted growth characteristics. Transition-acceleration experiments indicated that the initial microstructure before acceleration had a decisive influence on the final microstructure; only when the pre-transition microstructure contained no independently growing primary phase could a coupled eutectic structure be achieved at higher growth rates post-transition. Furthermore, directional solidification effectively improved both the strength and ductility of the Al-12%Ni alloy, with the elongation of the coupled eutectic structure obtained via transition acceleration showing further enhancement.

Full Text

Preamble

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MICROSTRUCTURE EVOLUTION OF DIRECTIONALLY SOLIDIFIED Al–12%Ni HYPEREUTECTIC ALLOY

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Abstract

The Al–12%Ni hypereutectic alloy (mass fraction) was prepared from pure Ni and Al (99.9%) by induction melting and directionally solidified at constant growth rates ranging from 1 m/s to 100 m/s. Experiments involving abrupt changes in growth rate were also conducted in a Bridgman-type furnace. After solidification, samples were rapidly quenched into liquid Ga–In–Sn alloy to preserve the microstructure. Microstructural observation using optical microscopy (OM) and scanning electron microscopy (SEM) revealed that at a growth rate of 1 m/s, the primary Al$_3$Ni phase disappeared after a certain growth distance, enabling coupled eutectic growth. When the Al$_3$Ni phase served as the leading phase at growth rates from 2 m/s to 100 m/s, its morphology was faceted. Experiments with abrupt growth rate changes demonstrated that the initial microstructure prior to the change determined the subsequent microstructure. Only when no coarse primary Al$_3$Ni phase existed before the abrupt change could a fully coupled eutectic structure be obtained at relatively higher growth rates. After an abrupt increase in growth rate, the growth of primary Al$_3$Ni phase was suppressed, allowing the coupled eutectic to grow continuously without coarse primary phases. Directional solidification effectively improved both the strength and plasticity of the alloy. Additionally, the elongation of Al–12%Ni alloy could be greatly enhanced through abrupt changes in growth rate during directional solidification.

KEY WORDS eutectic alloy, directional solidification, microstructure evolution, solidification mechanism

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1. Introduction

Eutectic alloys have attracted significant attention for their potential to form in-situ composites with excellent mechanical properties through directional solidification. The Al–Ni eutectic system is particularly promising due to its high strength and good thermal stability. However, the hypereutectic Al–12%Ni alloy typically solidifies with a coarse primary Al_3Ni phase that deteriorates mechanical properties. Controlling the solidification process to achieve a fully coupled eutectic structure is therefore crucial for optimizing performance.

Previous studies have shown that growth rate significantly influences microstructure evolution in directionally solidified alloys. The competitive growth between primary phases and eutectic structures depends on the solidification parameters, including temperature gradient (G) and growth rate (v). Understanding these relationships is essential for producing high-quality eutectic composites.

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2. Experimental Procedures

The Al–12%Ni alloy was prepared from high-purity Al (99.99%) and Ni (99.99%) by induction melting under an argon atmosphere. Directional solidification experiments were conducted in a Bridgman-type furnace with a temperature gradient of approximately 20 K/mm. Cylindrical samples (25 mm diameter) were placed in alumina crucibles and heated to 1070 K for 30 minutes to ensure homogenization.

Two types of experiments were performed:

1. **Constant growth rate experiments:** Samples were solidified at constant withdrawal rates of 1, 3, 5, 10, 15, and 100 m/s for distances exceeding 21.6 mm, then quenched into liquid Ga–In–Sn alloy to preserve the solid/liquid interface.
2. **Abrupt growth rate change experiments:** Samples were first solidified at an initial rate (v_0) for a distance (l_0), then the rate was abruptly increased to a higher value (v_1) and maintained for distance (l_1) before quenching. The experimental parameters are summarized in Table 1 .

Microstructural characterization was performed using Olympus GX71 optical microscope and Quanta 200FEG scanning electron microscope equipped with energy-dispersive spectroscopy (EDS). The volume fractions of phases were measured using Image-Pro Plus 6.0 software. Tensile tests were conducted on an Instron 5500 machine at a strain rate of 1$\times$10^{-3}$ s^{-1} using specimens with gauge dimensions of 10 mm length and 3 mm diameter.

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3. Results and Discussion

3.1 Microstructure at Constant Growth Rates

Figure 1 [Figure 1: see original paper] shows typical microstructures of directionally solidified Al–12%Ni alloys at different growth rates. At v = 1 m/s, the primary Al_3Ni phase disappeared after an initial transient region, resulting in a fully coupled eutectic structure (Fig. 1a). The white and black arrows indicate the initial solid/liquid interface and the quenched interface, respectively.

At higher growth rates (v = 2–100 m/s), the Al_3Ni phase appeared as faceted primary crystals distributed within the eutectic matrix (Figs. 1b and 1c). The morphology of the solid/liquid interface changed from planar to cellular as the growth rate increased (Fig. 2 [Figure 2: see original paper]). At v = 1 m/s, the interface was nearly planar with a smooth eutectic front (Fig. $2a_1$, a_2). At v = 2 m/s, faceted Al_3Ni crystals protruded into the liquid (Fig. $2b_1$, b_2). At v = 10 and 100 m/s, the interface became more irregular with pronounced faceting of the Al_3Ni phase (Figs. $2c_1$–d_2).

3.2 Effect of Abrupt Growth Rate Changes

Figure 3 [Figure 3: see original paper] illustrates microstructures before and after abrupt growth rate changes. In sample A (v_0 = 1 m/s, l_0 = 21.6 mm; v_1 = 2 m/s, l_1 = 10 mm), no primary Al_3Ni phase was observed either before or after the rate change (Figs. $3a_1$, a_2). The coupled eutectic structure was maintained throughout.

In sample B (v_0 = 2 m/s, l_0 = 25 mm; v_1 = 10 m/s, l_1 = 10 mm), coarse primary Al_3Ni crystals formed during the initial stage (Fig. $3b_1$). After the abrupt rate increase, these primary phases persisted and were not eliminated (Fig. $3b_2$). This demonstrates that the pre-existing microstructure determines the post-change microstructure.

In sample C (v_0 = 1 m/s, l_0 = 14.4 mm; v_1 = 10 m/s, l_1 = 10 mm), where the initial stage produced a coupled eutectic, the higher growth rate did not re-nucleate primary Al_3Ni. Instead, the fine eutectic structure continued to grow (Figs. $3c_1$, c_2). This confirms that suppressing primary phase formation during the initial stage is critical for achieving fully coupled eutectic structures at higher rates.

3.3 Mechanical Properties

Tensile test results (Table 2) show that directionally solidified samples exhibit significantly improved strength and ductility compared to as-cast material. Samples solidified at 1 m/s (fully eutectic) showed the best combination of properties. The elongation was further improved in samples subjected to abrupt growth rate changes, particularly when the initial low rate produced a defect-free eutectic structure.

3.4 Solidification Mechanism Analysis

The suppression of primary Al_3Ni phase at low growth rates can be explained by constitutional undercooling. The critical condition for planar eutectic growth is given by:

$$(cid:31)\text{"@'}i(cid:30)YZMNOP(cid:21)(cid:22)_3Q(cid:23)(cid:24)(cid:25)(cid:26)(cid:27)(cid:28)(cid:29)(cid:30)(cid:31)!\text{"}\#$$

where G is the temperature gradient, v is the growth rate, C_0 is the alloy composition, C_E is the eutectic composition, m is the liquidus slope, and D is the diffusion coefficient. For Al–12%Ni, using G = 20 K/mm, D = 0.62$×10^{-9}$ m^2/s, m_{Al}$_3$Ni = 11.6 K/%, C_0 = 12% Ni, and C_E = 6% Ni, the critical growth rate for planar eutectic growth is calculated to be approximately 1.5 m/s. This agrees well with experimental observations where v = 1 m/s produced a fully coupled eutectic.

The faceted morphology of Al_3Ni is consistent with Jackson's criterion. The α-factor for Al_3Ni is:

$,Tv0 = 1\mu m/sZ"g,j```Al3Ni(cid : 30);T8`(cid : 29)(cid : 30)l0 = 14.4mm,8(cid : 31)MNOPQR(cid : 21)(cid : 22)_1QR,! + (cid : 28)\#Al–12$

where ΔS is the entropy of fusion (33.45 J/(K $\cdot$ mol)) and R is the gas constant. The calculated $\alpha > 2$, indicating faceted growth, which matches the observed morphology at rates ≥ 2 m/s.

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4. Conclusions

1. At a growth rate of 1 m/s, the primary Al_3Ni phase in directionally solidified Al–12%Ni alloy disappears after an initial transient, resulting in a fully coupled eutectic structure.
2. At growth rates from 2 to 100 m/s, the Al_3Ni phase grows as faceted primary crystals, and the solid/liquid interface becomes increasingly cellular.
3. The microstructure before an abrupt growth rate change determines the final microstructure. Only when no coarse primary Al_3Ni exists prior to the change can a fully coupled eutectic be maintained at higher rates.
4. Abrupt growth rate changes effectively suppress primary phase formation and promote continuous coupled eutectic growth, significantly improving both strength and elongation.

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