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# Morphological instability of lamellar structures in directionally solidified Ni–Ni<sub>3</sub>Si alloys



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#### ABSTRACT

The morphological instability of lamellar structures in Ni–Ni<sub>3</sub>Si eutectic and hypereutectic alloys directionally solidified at low growth rates was investigated. The first instability in large lamellar structures was zigzag instability, which formed curved lamellae. A zigzag pattern was first displayed in three dimensions. The diffusion-limited growth of the Ni<sub>3</sub>Si phase decreased phase width and spacing, consequently causing zigzag instability. The reduced spacing was observed at  $\lambda/\lambda_{ave} = 0.9$ . After zigzag instability, the microstructure of the eutectic alloy turned into a labyrinth structure and lamellar fragmentation. However, in hypereutectic alloys, shape transition from lamellae to rods occurred, in turn, by the broken lamellae or elongated rods to dumbbell-shaped rods, peanut-shaped rods, and circular rods.

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### **1. Introduction**

Eutectic composites are among the most common two-phase microstructures in metallic alloys [1]. The two regular forms of eutectics are lamellar and rod eutectics. Within a finite growth rate range, the directional solidification of eutectic alloys at near-eutectic compositions usually yields lamellar or rod composite structures which have been regarded as high strength materials for the application at high temperatures such as turbine blades [2–4].

At a finite growth rate range, the selection and stability of either rod or lamellar eutectic in directional solidification depend on the volume fraction of the minor phase *f* and on the relative interface energy contributions; this dependency is well described by the Jackson–Hunt theory [5,6]. Lamellar eutectic growth is generally stable only over a finite range of lamellar spacing, and lamellar spacing adjustment in three-dimensions is through lamellar termination for small spacings and lamellar creation for large spacings [7]. Lamella termination instability occurs at a lamellar spacing of approximately 0.7  $\lambda_m$ , where  $\lambda_m$  is the minimum undercooling spacing. New lamellae are created to reduce large lamellar spacings after zigzag instability first occurs, and stable zigzag patterns, as well as disordered labyrinths, can form [8,9]. Zigzag instability, which occurs at a spacing of approximately 0.85  $\lambda_m$ , limits the growth rate range of lamellar structure production and is the only instability observed in transparent  $CBr_4-C_2Cl_6$  organic alloys with large spacing [8].

The occurrence of zigzag instability leads to the emergence of new growth patterns. The quantitative three-dimensional phase-field method was recently applied to simulate the instability process, thus providing new insights into the problems of microstructural formation and morphological instability [10,11]. The simulation results show that zigzag instability first has to occur for the development of large spacings and, depending on initial spacing and the volume fractions, leads to the breakup of lamellae into rods or labyrinthine structures [12–16]. The existence of the lamellar/rod transition has been proven in the Al-Cu system, which has a maximum stable spacing of 1.2  $\lambda_m$  [17]. However, the labyrinthine structures and curved lamellar structure (zigzag pattern) are not involved in the research of practical engineering materials, and the fundamental understanding of the morphological instability remains incomplete.

The aim of this paper is to present detailed experimental studies on the engineering Ni–Si alloy to systematically characterize the instability of lamellar structures at low growth rates. Regular lamellar structures have been obtained in directionally solidify Ni–Ni<sub>3</sub>Si eutectic alloys. Straight lamellar spacing can be increased by decreasing growth rate [18,19]. Presently, studies on morphological instability in lamellar Ni–Ni<sub>3</sub>Si alloy are rare. In this work, the lamellar structures for the eutectic and hypereutectic components were obtained at a certain growth rate. By further decreasing



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growth rate, the instability of lamellar structure will occur at large spacing. Furthermore, the dynamic process of lamellar instability was discussed.

# 2. Experimental procedures

Binary Ni–Si alloy ingots with nominal compositions of Ni– 11.5% wt. Si eutectic, Ni–11.8% wt. Si hypereutectic were produced by melting commercially pure Ni (99.99 wt%) and high-purity Si (99.9999 wt%) in a vacuum-induction melting furnace. Then, ascast rods were cut from the eutectic and hypereutectic ingots through spark machining. Rods were cut with diameters of ~4 mm and lengths of 100 mm to fit into alumina crucibles. Eutectic and hypereutectic rods were directionally solidified in a vertical Bridgman-type facility with induction heating at a thermal gradient of 250 °C/cm. The growth rates of eutectic alloys ranged from 0.5  $\mu$ m/s to 20  $\mu$ m/s, whereas those of hypereutectic alloys increased from 0.5  $\mu$ m/s to 4  $\mu$ m/s.

As previously described [20], after directional solidification, transverse sections were cut from specimens, mounted, polished, and subjected to selective electrochemical dissolution in an aqueous electrolyte containing 6 wt% ammonium sulfate, 1 wt% tartaric acid, 6 wt% citric acid, and 2 vol% glycerol. The microstructure of the selectively dissolved specimen was observed under a scanning

electron microscope (JEOL model JSM-6390A) equipped with an energy-dispersive X-ray analyzer.

# 3. Results and discussion

Fig. 1 shows the microstructure of the directionally solidified Ni-Ni<sub>3</sub>Si eutectic grown in this study after selective dissolution for 68 h. At growth rates of 4–20 µm/s, a fully eutectic structure without any dendritic regions was observed throughout the specimen. The black stripe corresponded to the  $\alpha$ -Ni phase, whereas the gray stripe corresponded to the  $\beta$ -Ni<sub>3</sub>Si phase. At the growth rate of  $4 \mu m/s$  (Fig. 1[a]), the lamellar eutectic structure appeared wavy and straight, and a large proportion of wavy lamellae were present throughout the entire transversal section of the specimen. Two kinds of morphologies intersected at the grain boundary. Waviness and the curvature frequency, direction, and degree of the wavy lamellar structure were identical in one grain. Given that produced larger lamellar spacing under decreasing growth rate, the lamellar structure underwent zigzag instability, which is a transition from straight to wavy lamellae. Zigzag instability did not occur drastically but occurred over a range of growth rates in polycrystalline structure where both mixed morphologies are present. This can be attributed to the presence of a range of spacing in the polycrystalline structure.



Fig. 1. Microstructure of Ni–Ni<sub>3</sub>Si eutectic alloys at different growth rates: (a) 4, (b) 6, (c) 8, (d) 10, (e) 12, (f) 15, and (g) 20  $\mu$ m/s.

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