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Grain refinement of superalloy K4169 by addition of refiners: cast structure and refinement mechanisms

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Abstract

Grain size and microstructural features of cast superalloy K4169 were investigated under various melting and casting conditions with and without the addition of grain refiners. It is found that lowering pouring temperature and adding refiners to the melt can lead to grain refinement of γ matrix and improve the proportion of equiaxed grains. At a conventional pouring temperature of 1400 °C, the average size of equiaxed grains could be refined to the order of ASTM 3.2, the proportion of equiaxed grains at transverse cross-section could be improved from 56 to 99%. The results also indicate that the average length of primary dendrite axis is shortened with the addition of refiners, but the secondary dendrite arm spacing keeps almost unchanged because local solidification time remained constant. In addition, the microsegregation of main elements such as Fe, Cr, Nb, Mo and Ti is alleviated with the decrease in grain size, and the grain morphology have transformed from dendrite in coarse- to granulate in fine-grained castings. At higher melt pouring temperature, the amount of microporosity in samples with the addition of refiners can be greatly reduced. The mechanisms of grain refinement and increase in equiaxed grain proportion were proposed.

Keywords: Superalloy; Grain refinement; Solidification; Dendrite structure; Microsegregation; Grain size; Microporosity

1. Introduction

Gas turbines are now widely used in a variety of aircraft, marine, industrial and vehicular applications as well as high performance military aircraft. Since early 1960s, a trend of rapidly rising turbine inlet temperature has occurred because of increase in efficiency. The growing demands of advanced gas turbine engine technologies have required the development of high strength heat resistance materials. Various approaches have included directionally solidified alloys and eutectic, single crystal alloys, superalloy powder metallurgy, oxide dispersion strengthening as well as processing improvement to keep pace with increasing inlet temperature [1–3].

On the other hand, the operating temperature of turbine wheels of aircraft engines and industrial turbine engines is relatively low. For example, a rotor is customarily divided into three general areas, the hub, the rim and the blades. In the hub section, the operating temperature is approximately 700 °C [4], which is typically below the creep range. Whereas stresses from centrifugal loads are high. High tensile strength and good low cycle fatigue (LCF) resistance are primarily required. In this range, a uniform and fine grain size is desired to promote fatigue properties and resistance to crack growth.

Since 1970s, efforts have been made to replace to forged turbine wheels which have been plagued with large grains in the central hub areas because they are seldom adequately worked [5] with investment cast wheels. However, the predominant feature of conventional investment cast alloys is microstructural coarseness or non-uniformity of grain size. Therefore, it is of utmost importance to develop methods for

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obtaining fine and uniform grains in turbine wheels and other engine components.

2. A literature review of grain refinement of superalloys

Refinement of cast structure requires that nucleation occurs at a large number of sites and that extensive growth of crystals be avoided. It follows that grain refinement necessitates both ease of nucleation and inhibition of the continued growth of crystallites in the melts. The existing knowledge of grain size control can be divided in three basic ways: mechanically, chemically or thermally. That is agitation of the melt during the freezing, nucleation promoting and growth hindering additions or rapid cooling.

In the early 1980s, the first generation of fine-grain casting technique named Grainex® (GX) was developed by Howmet Turbine Component Corporation [6,7]. In this process, castings are agitated during solidification to shear dendrites and initiate multiple nucleation sites, which subsequently leads to an uniform refined grain size, ranging from ASTM M9–M13 (1.6–0.4 mm), close to ASTM 0 (0.36 mm). The GX process has been used to produce Rolls-Royce Corporation Model 250 first stage wheels [8].

In 1983, Howmet introduced second generation of fine grain casting techniques [7,9–11]. This process is referred to as Microcast-X® and offers the potential for producing integrally cast rotor components with a grain size in the ASTM 3–5 range (125–65 μm). In comparison with GX castings, which exhibit a dendritic structure, the Microcast-X® microstructure exhibit a cellular type of structure. Basically, the process of Microcast-X® involves casting molten metal having low pouring temperature within about 10 °C before the melting point into a heated mould. During pouring, turbulence in the molten alloys increases its surface-to-volume ratio and, in turn, the heat-extraction rate. The closely controlled, low pouring temperature and high heat-extraction rate are primarily responsible for the cellular, forging-like microstructure. At the same time, AiResearch Casting Company [12] developed a kind of fine grain casting process (FGP). The process was established to use low pouring temperature (melting point +22 °C), low melt superheat temperature, moderate mould preheating temperature (1100 °C) and local chills. For the IN 713 LC and MAR-M 247 alloy, grain size in the range of ASTM 1-2 (0.25–0.18 mm) was obtained. The similar results have been obtained for In-713LC alloy recently [13]. Like FGP, Microcast-X® process also reduces molten-metal fluidity, and has less ability to fill thin sections. Furthermore, dispersed microporosities within castings due to low poring temperature must be eliminated by hot isostatic pressing (HIP) process, which increases in production costs.

An alternative method of refinement of cast structure is nucleation promoting and growth hindering additions before pouring, which is more effective for grain refinement. The criteria that good inoculant or refiner must process to perform as a stable substance for heterogeneous nucleation, including stability in the melt, be finely dispersed, have similar density to the melt and be able to initiate freezing at very small undercooling [14]. These principles have been used successfully to refine a considerable range of alloys either by deliberate additions to the melt or by coating the interior surface of investment mould [15–17]. Unlike most of the nonferrous alloys, grain refinement of superalloys during cast process is more difficult due to their complicated composition, multi-phases, high melting point and melted in vacuum condition.

Since 1980s, investigations on grain refinement for superalloys by chemical method (addition of refiners) have been performed by researchers. Although additions of boron [18], particles of metallic oxides [19], refractory carbides and nitrides [20] could refine grains, they were not used in the industrial scale due to inclusion or lowering the incipient melting temperature of the alloys.

Although it has been considered that the inoculation of the melt is not often used with superalloys for their high-inclusion sensitivity, some successful refinement processing was still related with this method [21]. The key to the argument is how to select the proper inoculants, which have high effectiveness to promote nucleation and do not produce the inclusion.

In the present study, two intermetallic compounds from the ternary system Co–Fe–Nb and Cr–Mo–Nb were chosen as inoculants, and their effects on the grain structures of cast superalloy K4169 under various melting and casting conditions were investigated.

3. Materials and experimental procedures

3.1. Preparation of refiners

According to the above-mentioned principles for grain refinement by chemical method, ideal refiners for superalloys should have the following characteristics:

(1) They are composed of the alloying elements in superalloys, such as Fe, Ni, Nb or Cr.
(2) Their melting point should be higher than 1400 °C, the temperature that the alloy is solidified.
(3) The difference of density between refiner and the alloy should be as small as possible, in order to avoid float or deposition. Accordingly, the density of refiner should be controlled between 8.0–9.0 g/cm^3.
(4) Good lattice matching between refiner and the alloy should be required to decrease the interfacial energy between them. Generally, if lattice misfit between refiner and alloy being solidified is smaller than 10%, the refiner can act as an effective nucleation substrate [22].
(5) Its must be brittle and easy to triturating.

Base on calculation of lattice misfit and extensive experiments [23,24], we found that some high melting intermetallic compounds satisfied above conditions simultaneously. The button ingots of refiners were prepared by melting an appro-