

Effect of minor alloying additions on the carbide morphology in a single crystal Ni-base superalloy

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Received 10 July 2007; revised 6 September 2007; accepted 7 September 2007
Available online 26 October 2007

Carbon and boron have been added to single crystal Ni-base superalloys to increase the yield and defect tolerance, but the effect of these additions on the solidification is not well understood. Carbon was added to the second-generation single crystal Ni-base superalloy, CMSX-4, to form MC-type carbides. Nitrogen and boron were also added in an attempt to alter the carbide morphology. This investigation examines the effect of these alloying additions on the carbide morphology.

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Keywords: Nickel alloys; Directional solidification; Carbides

Although initially removed from single crystal alloys, carbon additions have been shown to significantly reduce the frequency of solidification defects [1,2] and to reduce the formation of surface scale on single crystal nickel-base superalloy castings [3]. Initially, this decrease in the frequency of defects was attributed to reduced solidification segregation [1,2]. However, more recent results clearly indicate that the carbon addition blocks fluid flow due to the formation of carbides in the interdendritic region [4,5]. Carbon additions have also been shown to dramatically reduce scale formation during casting [6]. The morphology of the primary MC-type carbides that form in carbon-containing single crystal Ni-base superalloys would be expected to have an impact on the fluid flow and defect formation during solidification of the single crystals. However, the effect of the alloy content on carbide morphology has not yet been reported.

Nitrogen, whose content can be high in reverb, can result in an increase in microporosity [7,8]. A high nitrogen content leads to the precipitation of TiN particles throughout the mushy zone, which provides a heterogeneous nucleation site for carbides. Heterogeneous nucleation causes the carbides to precipitate at a higher temperature and higher position in the mushy zone than in the case of homogeneous nucleation. Therefore, these

nitrides may have an impact on the defect formation during solidification [9]. The effect of TiN on changing the precipitation temperature of carbides is reduced as the withdrawal rate increases due to the increasing prevalence of homogeneous nucleation [7].

Boron has been shown to reduce carbide size, another factor that may affect defect formation during solidification [10,11]. The reintroduction of both carbon and boron into single crystals has proven beneficial in reducing solidification defects and improving mechanical properties [6,12–15]. However, the effect of boron additions on the carbide morphology in single crystal Ni-base superalloys has not been fully characterized.

The solidification rate has a large effect on carbide size and morphology, with higher rates producing finer carbides [7]. High solidification rates and small thermal gradients also result in more faceted nodular carbides, due to their lower energy configuration [16]. Carbide shapes are related to their formation temperature because this determines their position in the mushy zone. Higher carbide solvus temperatures result in carbides forming at higher positions within the mushy zone and are not restricted by fully formed dendrite arms. This allows the carbides to form blocky, low surface energy structures [7]. It should also be noted that the morphology of the carbides may also impact the primary dendrite arm spacing, the ability of the resulting carbides to block fluid flow during solidification and, therefore, the effectiveness of the carbon addition on reducing solidification defects.

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Hafnium additions, which increase the carbide precipitation temperature, have also been shown to change the carbide morphology from script to nodular in a powder metallurgy superalloy [17]. Tantalum and niobium additions to superalloys alter carbide composition, but not morphology, since they do not significantly impact solidification temperature [18].

The single crystal samples used in this study were from a single master heat of CMSX-4 (Ni–9.6Co–6.5Cr–6.4W–2.9Re–6.5Ta–6.0Al–1.0Ti wt.%) and processed at PCC Airfoils (Minerva, OH). One baseline heat without any intentional alloying additions and three alloy modifications were cast from the same master heat (Table 1). A carbon level of 0.05 wt.% was selected for all of the carbon-containing alloys in this study. The minor alloying element modifications used in this study were the addition of carbon (C), carbon and boron (C + B), and carbon and nitrogen (C + N). All additions were made just prior to pouring. The carbon and boron additions were made by adding the appropriate amount of graphite and/or boron powder wrapped in Ni-foil. The nitrogen addition utilized high-purity CrN powder wrapped in Ni-foil. Each 12 kg charge was investment cast into a mold with 20 12.5 cm long bars with a 1.25 cm diameter. These bars were processed using a Bridgman-type directional solidification furnace using helical selectors to produce single crystal samples with the [001] orientation. All molds utilized a withdrawal rate of 20 cm h⁻¹, resulting in a thermal gradient estimated at 30–40 °C cm⁻¹. Macroetching was used to insure that the samples used in this study were single crystals.

Optical microscopy was used to examine the microstructures and measure primary dendrite arm spacings (PDAS) and carbide morphologies of metallographically prepared, polished and etched (Pratt & Whitney Etch 17: 100 ml H₂O + 100 ml HCl + 100 ml HNO₃ + 3 g MoO₃) samples in the as-cast condition.

Scanning electron microscopy (JEOL 6400) was used to examine microstructural features in detail. Most micrographs were taken from secondary electron imaging on etched microstructures. Backscattered imaging was used on unetched samples to determine elemental segregation and to examine precipitates. An electron microprobe (JEOL 733) was used to quantitatively identify the compositions of primary carbides.

Carbide morphologies of the as-cast samples of CMSX-4 were examined by deep etching of transverse and longitudinal samples. A solution of 30% hydrogen peroxide was mixed with 70% hydrochloric acid. This technique removed much of the γ and γ' , leaving the carbides exposed. The deep etched samples were then examined by SEM.

Dendrite arm spacing was determined by the square root of the number of primary arms in a given transverse area. The carbide and eutectic volume fractions were determined using manual point counting using backscattered SEM micrographs at a magnification of 2000x.

Differential thermal analysis (DTA) was performed at Dirats Laboratories (Westfield, MA). Samples weighing at least 250 g were heated at a rate of 20 °C min⁻¹ and compared with a reference of pure Ni. The transformation temperatures were obtained using on-heating data to avoid any effects of supercooling [19].

The carbides were observed in the interdendritic regions, but were visible only at higher magnifications (Fig. 1). At low magnification, all of the variations of CMSX-4 samples appear to have a similar microstructure. The change in dendrite morphology for the boron and nitrogen addition samples was observed only at higher magnifications. The addition of C to CMSX-4 resulted in a reduction in eutectic fraction, and the formation of carbides that precipitated between the secondary arms and, in some cases, appeared to block the development of some tertiary arms. The carbon and boron additions also exhibited a similar carbide structure. However, the carbides in the carbon + boron alloy were more blocky and irregular in shape and had a less uniform distribution. The addition of carbon also increased the PDAS (Table 2), similar to previous reports [4], due to the less regular structure of primary dendrite arms. However, the increased spacing was not seen in the two samples containing carbon and boron (C + B) and carbon and nitrogen (C + N).

The addition of carbon to the alloy decreased the solidus and liquidus slightly in the DTA testing (Table 2). The addition of carbon and boron led to the largest reduction in the as-cast solidus. The boron-containing alloy also had the lowest carbide solvus in the as-cast condition. The transformation temperatures observed in the carbon and nitrogen alloy were very similar to those of the carbon only alloy. However, it should be noted that the differences in all of these transformation temperatures (Table 2) are limited due to the low level of the alloying additions used in this study. Furthermore, the carbide precipitation temperatures of the boron and nitrogen-containing alloys are lower than the carbon only alloy, which indicates that the carbides in the boron and nitrogen-containing alloys are forming lower in the mushy zone.

Deep etching of longitudinal samples revealed differences between the dendritic carbide structures in the CMSX-4 variants (Fig. 2). In the CMSX-4 with only the 0.05C addition, the carbides were observed to be interconnected networks of carbide sheets which grew in tandem with the dendritic structure. In the CMSX-4

Table 1. Composition of CMSX-4 alloy modifications

CMSX-4 sample ID	Carbon aim (wt.%)	Carbon actual (wt.%)	Boron aim (wt.%)	Boron actual (wt.%)	Nitrogen aim (ppm)	Nitrogen actual (ppm)
Baseline	0.000	0.002	ALAP ^a	0.0004	ALAP ^a	2
C	0.050	0.066	ALAP ^a	0.0011	ALAP ^a	3
C + B	0.050	0.063	0.005	0.0068	ALAP ^a	3
C + N	0.050	0.063	ALAP ^a	0.0011	25	23

^a ALAP – as low as possible.

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