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Effect of solidification rate on MC-type carbide morphology in single crystal Ni-base superalloy AM3

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Abstract: In order to study the effect of the withdrawing rate on carbide morphology, MC-type carbide in single crystal superalloy AM3 was systematically investigated with sample growth rates from 3.5 μm/s to 500 μm/s. The carbide morphologies were investigated by scanning electron microscopy (SEM), and the electron probe microanalysis (EPMA) was used to characterize the carbide composition. The results indicate that the solidification rate is the important factor governing MC carbide growth morphology, size and distribution, composition and growth mechanism. With the increase of withdrawing rate, nodular, rod-like, Chinese script types of carbide morphology are observed. For the low withdrawing rate, with the increase of withdrawing rate, the carbide size becomes larger. For the case of dendritic interface, the carbide size becomes smaller with refinement of dendrites as withdrawing rate increases. The volume fraction of carbides increases with the withdrawing rate increasing.

Key words: single crystal; Ni-base superalloy; carbide morphology; withdrawing rate

1 Introduction

Carbides of the MC-type formed during solidification of nickel-base superalloys contribute to strengthening of grain boundaries at elevated temperatures. This strengthening role seems to depend significantly on carbide geometry. Its volume fraction, size and morphology have a very important effect on the mechanical properties and solidification behavior of nickel-base superalloys[1−6]. Numerous investigations [7−10] have characterized the formation sequence, composition and morphological evolution of this important carbide, in different (from slow to rapid) solidification conditions. BALDAN[7] investigated the effects of solidification parameters on the morphology of MC carbides in DS200 alloy. The shape of MC precipitates, however, remained almost unchanged with growth rate[11−12]. The solidification rate has a large effect on carbide size and morphology, with higher rates producing finer carbides[8]. The results indicate that the processing parameters such as sample growth rate, thermal gradient, solidification interface shape and chemical composition are important factors governing MC-carbide growth morphology (size, distribution and composition) and growth mechanism. It is now well understood that near-equilibrium growth morphology of MC-carbides in superalloys is octahedral blocks, which gradually transform to Chinese script morphology as the carbide growth rate increases. However, all of the above researches were related to directionally solidified superalloy and less research reported about single crystal superalloy and carbide morphology under high thermal gradient. Only FERNANDENS et al[13] investigated the effects of solidification parameters on the morphology of MC carbides in IN100 single-crystal. It was observed that high solidification rates and lower thermal gradients resulted in more faceted nodular carbides, due to their lower energy configuration. Moreover, carbide formation requires further investigation, addressing relationships among processing conditions, chemical composition, morphology and growth dynamics, such as nucleation and growth behavior. The purpose of this work is to further correlate withdrawing rate with type, shape, size

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and composition of MC carbide in AM3 single crystal superalloy directionally solidified under high thermal gradient.

2 Experimental

Table 1 lists the nominal composition of the alloy. The single crystals were cast in the [001] orientation by using a bottom seeding technique during directional solidification. The high thermal gradient up to 300−400 K/cm was achieved by heating the samples using dual-resistance and cooling them by low melting liquid metals simultaneously. Six withdrawing rates of 3.5, 10, 50, 100, 200, and 500 μm/s were used. The orientation of each single crystal sample with a misorientation of greater than 12° was considered defective and was not utilized in this work.

Table 1 Composition of modified AM3 used in this study (mass fraction, %)

Ni	C	Cr	Co	Mo	W	Al	Ti	Ta
Bal.	0.15	7.85	5.47	2.30	5.03	6.00	2.09	3.56

ZEISS SUPRA 55 field emission scanning electron microscope was used to examine the distribution and morphology of carbides in the specimens which were prepared via mechanical polishing followed by ultrasonic cleaning. Carbide morphologies were observed with back-scattered electron imaging. The transverse sections of the samples were electro-etched using 17% nitric acid, 33% hydrofluoric acid and 50% glycerin solution to remove the matrix and reveal the stereo carbide structure.

Carbide composition was determined by WDS method of electron probe microanalysis (EPMA) on polished and un-etched samples, using a JEOL model superprobe JXA-8100 operated at 15 kV with a beam size of 0.5 μm. The carbide volume fraction was determined using manual point counting using back-scattered SEM micrographs.

3 Results and discussion

Figure 1 shows the longitudinal micrographs at different withdrawing rates in the alloy. Planar interface is observed at withdrawing rate of 3.5 μm/s. The evolution of interface morphologies changes in the

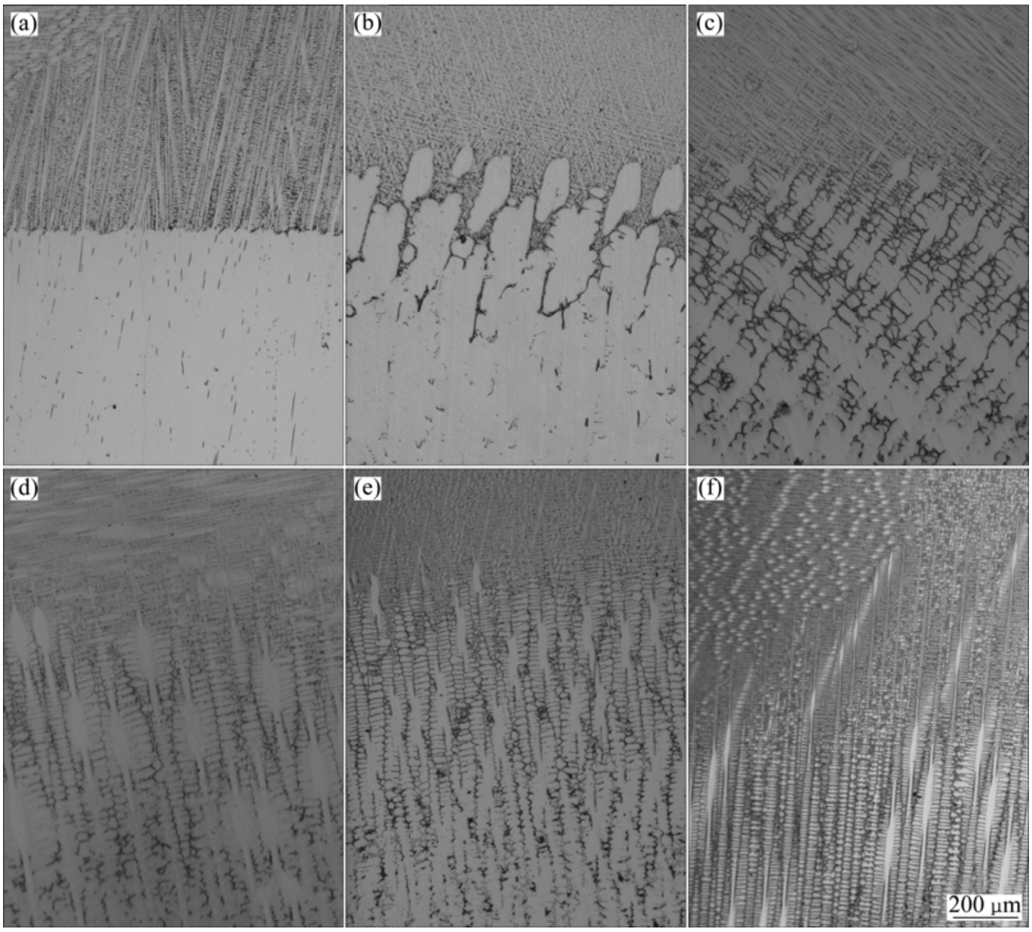

Fig.1 Growth morphologies at various withdrawing rates: (a) Planar, 3.5 μm/s; (b) Cellular, 10 μm/s; (c) Coarse dendrite, 50 μm/s; (d) Coarse dendrite, 100 μm/s; (e) Fine dendrite, 200 μm/s; (f) Fine dendrite-cellular, 500 μm/s

sequence of planar, cellular, coarse dendrite, dendrite and fine dendrite with increasing withdrawing rate (v) ranging from 3.5 to 500 μm/s, as shown in Figs.1(a)−(f). Cellular interface is observed at the withdrawing rate of 10 μm/s (Fig.1(b)), and coarse dendrite interface morphology can be observed at the withdrawing rate of 50 μm/s (Fig.1(c)). Dendrite interface morphology can be observed at withdrawing rates of 100 μm/s and 200 μm/s (Figs.1(d) and (e)). When the withdrawing rate increases to 500 μm/s, the interface morphology changes to fine dendrite-cellular (Fig.1(f)).

Figure 2 shows the transverse morphologies of the carbides under different withdrawing rates. Several types of carbide morphologies are observed in the etched samples. The first type is nodular morphology (Fig.2(a)), which is found mainly in the alloy with withdrawing rate of 3.5 μm/s. The second type is rod-like morphology (Fig.2(b)). The third type of morphology found in other alloys is the blocky carbides (Fig.2(c)) which were reported to form at temperatures close to the liquidus[13]. The last type is a script-like morphology (Figs.2(d), (e) and (f)) which appear to grow from the center of the interdendritic region and form a cubic dendrite shape. The carbides exhibit orthogonal dendrite arms that grow in a sheet-like morphology and then separate into a rod-like structure. This type of morphology constitutes most of the carbides found in these samples.

At the withdrawing rate of 3.5 μm/s, the interface morphology of planner discrete carbides forms, most of the morphologies are nodular, and trace amount of rod morphology can be observed (Fig.3(a)). At withdrawing rate of 10 μm/s, the carbide morphology is mainly rod-like (Fig.3(b)), and trace nodular and blocky morphology can also be observed. When the withdrawing rate is increased to 50 μm/s, 100 μm/s, 200 μm/s and 500 μm/s, the thermal gradient is up to 300−400 K/cm, and the carbide morphology becomes a highly developed Chinese-script type (Figs.3(c), (d), (e) and (f)).

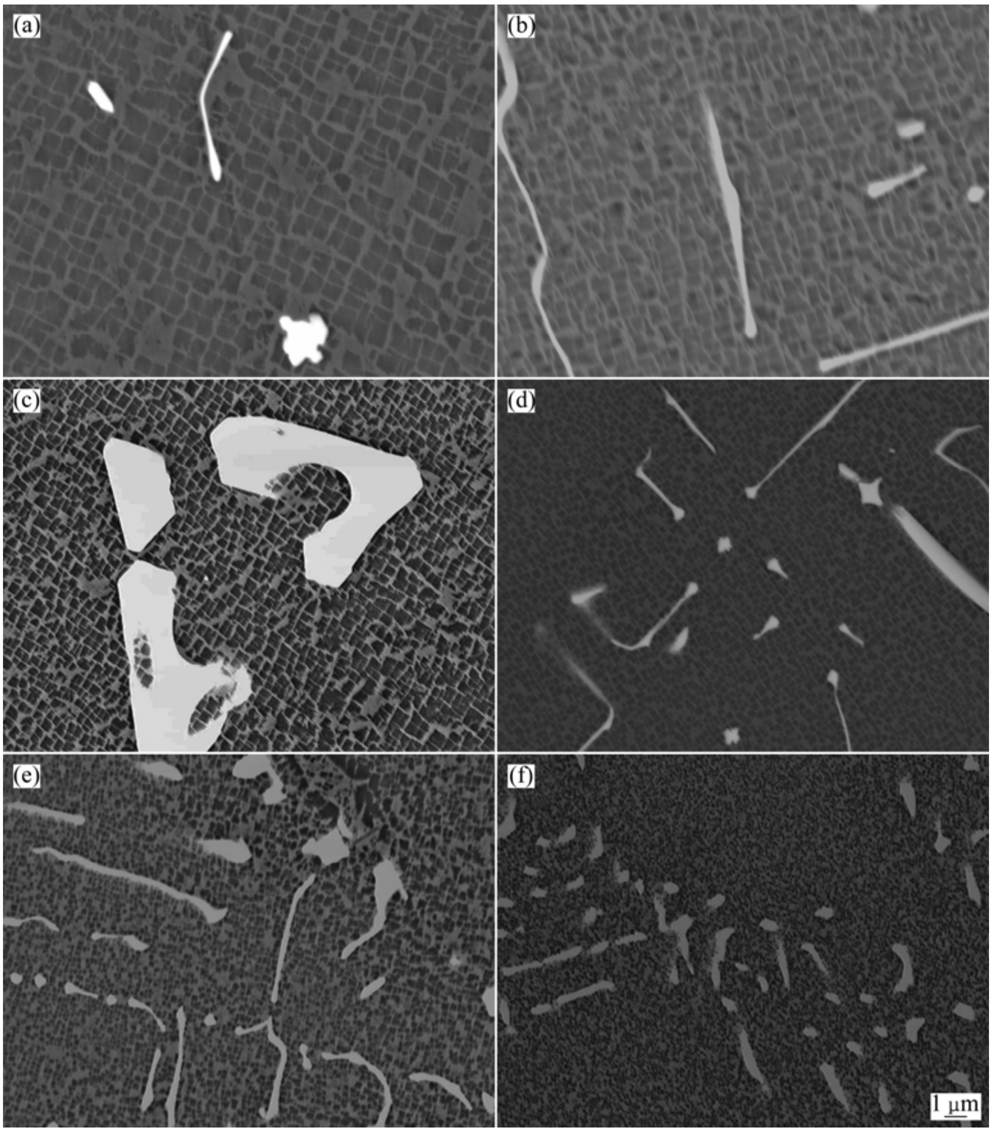

Fig.2 Carbide morphologies in transverse section of alloys at various withdrawing rates (in high magnification): (a) 3.5 μm/s; (b) 10 μm/s; (c) 50 μm/s; (d) 100 μm/s; (e) 200 μm/s; (f) 500 μm/s

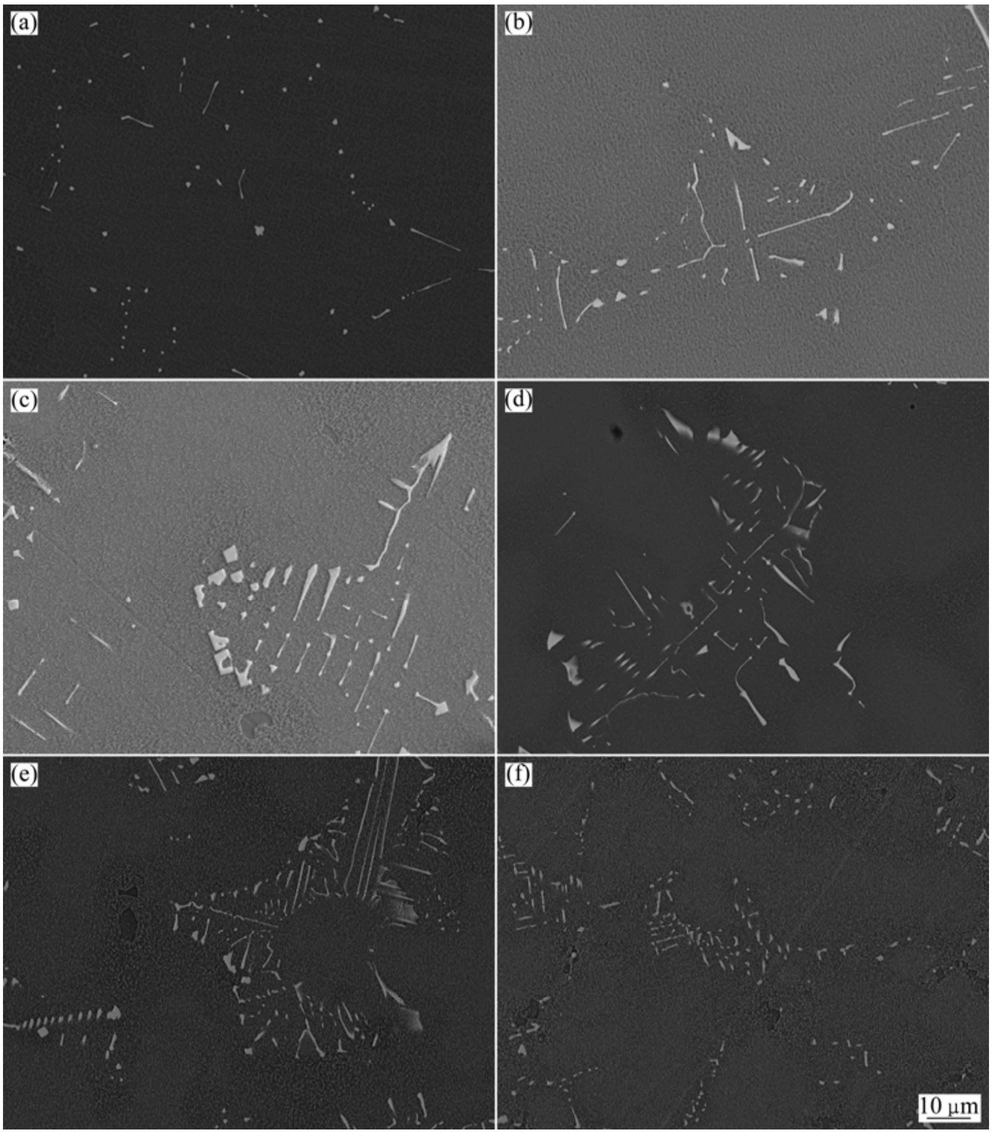

Fig.3 Carbide morphologies in transverse section of alloys at various withdrawing rates (in low magnification): (a) 3.5 μm/s; (b) 10 μm/s; (c) 50 μm/s; (d) 100 μm/s; (e) 200 μm/s; (f) 500 μm/s

Considering the relatively high withdrawing rates and high thermal gradient during the processing, the depth of the mushy zone and the time for solidification would be expected to be relatively small. As the matrix of γ and γ' form in the dendrite, the carbon and tantalum are rejected to the liquid and enriched in the liquid. The accumulation of carbide forming elements along with the high withdrawing rate and directional solidification results in the formation of a complicated carbide network in the alloy. The morphology of these types of carbides was also reported to be affected by the carbide precipitation temperature with respect to the liquidus and solidus temperatures of the alloy[13]. Script-like carbides of TaC were also formed in other nickel-base superalloys at a withdrawing rate of 18 cm/h[14].

The average size of carbide is often correlated with the withdrawing rate (Fig.4). As the withdrawing rate decreases, the time for diffusion increases, allowing additional time for carbide growth. When the withdrawing rate is low (3.5, 10 and 50 μm/s), carbides become coarser with withdrawing rate increasing; and when the withdrawing rate is high (100, 200 and 500 μm/s), with further increase of the withdrawing rate, the Chinese script-type carbide becomes finer. When the withdrawing rate is 3.5 μm/s, the interface morphology is planar; when the withdrawing rate is 10 μm/s, the interface is cellular; and when the withdrawing rate is 50 μm/s, the interface morphology is coarse dendrite. For the different interface morphology, the diffusion mechanism is different. But when the withdrawing rate is high (100, 200 and 500 μm/s), the interface morphologies are all dendrites. For the same interface morphology, the carbide size only depends on the time for diffusion. Beyond the peak, curve begins to descend as a function of withdrawing rate, but the variation is not so dramatic as that on the pre-peak side. This result is in agreement with the directionally solidified superalloy IN 738LC[15], and this result is in disagreement with the single crystal superalloy IN100[13]. In this investigated alloy of AM3, no Hf element is contained; but in IN100 containing trace amount of Hf element, there is HfC in IN100 superalloy. No carbide with size more than 3 μm can be observed in all alloys with different withdrawing rates. But in Ref.[13], carbide size can reach 30 μm, and this may be caused by the large temperature gradient. From this point, it can be concluded that the high temperature will be beneficial to mechanical properties.

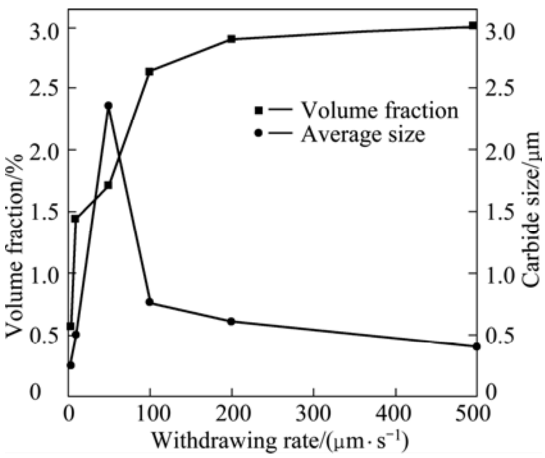

Fig.4 Volume fraction and average size of carbides in single crystal superalloy AM3 grown at different withdrawing rates

The volume fraction of carbide is a significant function of withdrawing rate. With the withdrawing rate increasing, the volume fraction of carbides increases. This result is in disagreement with the directionally solidified superalloy IN738 LC[15] and IN100[13]. And when the withdrawing rate reaches the maximum of 500 μm/s, the volume fraction of carbides reaches the maximum value (Fig.4).

The results indicate that the withdrawing rate is the important factor governing MC carbide growth morphology, size, distribution, composition and growth mechanism. It is now well understood that the near-equilibrium growth morphology of the MC carbide in single crystal superalloy AM3 is nodular, which gradually transforms to rod-like and Chinese script-type morphology as the withdrawing rate increases.

EPMA measurements were conducted on various types of carbides, as shown in Fig.5. A comparison of compositions of the various types of carbides is presented in Table 2. Measurements were made using the point counting technique and considering nickel to be the balance element in the carbide. The carbides are contained in a specimen grown at 500 um/s. Microprobe analysis of carbides shows that all types of carbide are rich in Ta, and blocky carbides also consist of Ti and W, with less amounts of Mo and some trace amounts of Co and Cr. Nodular carbide consists of W and Ti, with less amounts of Cr and Mo and some trace amounts of Co. Rod-like carbide consists of W and Cr, with less amounts of Mo, Co and Ti. These results suggest that the carbides are of MC-type, or TaC. It can be seen that blocky carbide is richer in titanium than the nodular and rod-like carbide, and nodular and rod-like carbides are richer in tungsten than the blocky carbide.

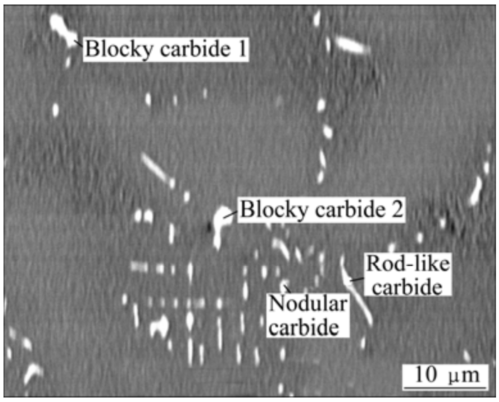

Fig.5 Various carbide morphologies by EPMA analysis

Table 2 Composition of various types of carbide in specimen grown at 500 μm/s (mass fraction, %)

Element	Blocky carbide 1	Blocky carbide 2	Nodular carbide	Rod-like carbide
Al	0.631	0.355	1.980	3.260
Ni	12.160	5.290	28.800	41.320
W	12.840	17.700	12.240	9.920
Co	1.160	0.562	2.480	3.630
Mo	6.790	5.490	4.990	4.210
Ta	40.920	46.230	29.670	20.970
Ti	14.630	14.590	9.590	2.690
Cr	3.630	0.960	5.370	6.370

Similar composition measurements were conducted on carbides in various specimens grown at different withdrawing rates. No significant variation of composition is found with growth conditions.

4 Conclusions

1) The morphology, size and amount of MC carbides in single crystal nickel-base superalloys are affected by the withdrawing rates. In directionally solidified alloys with planar interfaces, the carbides usually have a nodular appearance. A blocky type carbide precipitate is observed for growth with a cellular

interface. A highly developed Chinese script-type morphology can be found during dendritic growth.

2) For the low withdrawing rate, with increasing the withdrawing rate, the carbide size becomes larger. In the case of dendritic interface, the carbide size becomes smaller with refinement of dendrites as withdrawing rate increases. At withdrawing rate of 50 um/s, the maximum size of carbide can be observed in all of the alloys. The volume fraction of carbides increases with increasing the withdrawing rate.

3) EPMA analysis shows that all types of carbide are rich in tantalum. It can be seen that blocky carbide is richer in titanium than the nodular and rod-like carbide, and nodular and rod-like carbides are richer in tungsten than the blocky carbide.

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