HEAT TREATMENT, MICROSTRUCTURE, AND CREEP STRENGTH OF $\gamma/\gamma'-\alpha$ EUTECTIC DIRECTIONALLY SOLIDIFIED BY FLUIDIZED BED QUENCHING

Y.G.NAKAGAWA, A.OHOTOMO, Y.SAIGA
IHI RESEARCH INSTITUTE
M.NEMOTO, H.SUTO
TOHOKU UNIVERSITY

A laboratory evaluation of a directional solidification method by means of fluidized bed quenching(FBQ) in which zirconia particles fluidized by argon gas flow was used as a cooling medium was made in comparison with the high rate solidification (HRS) and the liquid metal cooling(LMC) method. Of the thermal analysis, the thermal gradient established in a solidifying ingot by FBQ and LMC was found to be about ten times as much as that by HRS. The microstructure of as-grown and heat treated $\gamma/\gamma'-\alpha$ composites made by FBQ was examined in conjunction with the hardness change and the creep rupture strength.
The as-grown structure was found to be formed by the eutectic followed by a peritectoid reaction which resulted in blocky γ' regions round the Mo fibers. Quenching the eutectic above the peritectoid temperature diminished the blocky γ'. The subsequent aging treatments induced small Mo platelets above 830°C and below this temperature a metastable Ni_2Mo particle precipitation in the γ phase causing appreciable composite hardening. The creep rupture strength, however, was reduced by the heat treatments presumably because of the dense quench-in dislocations in the matrix.

1. INTRODUCTION

Directionally solidified(DS) eutectics have received a considerable attension for past ten years as gas turbine blade materials with improved service capability over currently available columnar grained or single crystal blades.[1] Following the two most promising first generation eutectics, namely γ/γ'-δ and γ/γ'-MC for which further off growth axis ductility is desired, Ni-Mo-Al system basiclly composed of ductile reinforcing phase, Mo fibers, embedded in the ductile matrix (γ/γ') is considered to have a high engineering potential among those recently evaluated second generation eutectics.[2] Prior to alloy modification for desired blading material through alchemic combination of alloying elements, fundamental knowledge of the microstructure of the composites, and its high temperature behavior upon various heat treatments are need to be clarified.

The production of in-situ composites hangs on the commercial feasibility of a DS furnace having a thermal gradient, G, several times larger than the current high rate solidification(HRS) method in order to meet the full plane front growth condition. A process which is designed to offer a high gradient is the so-called low melting point metal cooling(LMC) in which the mold is withdrawn from the furnace and directly quenched in a molton metal of low melting point depriving heat at a far faster rate than by radiation.[3] The cooling efficiency, however, is determined by the heat content balance between the cooling medium and the castings; thus, the dimension of the quenching bath is required to be roughly proportional to that of the casting. It is also necessary to control the stirrer, heating and cooling systems, and the purity of the low melting point metal for achieving an uniform temperature distribution in the bath.

In the present study a laboratory evaluation of DS method by means of fluidized bed quenching(FBQ) was made in comparison of HRS and LMC method. The as-cast structure of γ/γ'-α eutectics made by FBQ, and solutionized(SHT) as well as successively aged structures of the system were also examined together with mechanical property changes. The paper describes some of the outcomes to date.

2. EXPERIMENTAL

In order to attain a DS method with a high temperature gradient, a fluidized bed was placed beneath a melting furnace of 1 kg charge capacity. The bed consisted of a solid particle bath with a built-in water cooled coil, and a gas-accumulator right under the bath. A sintered stainless steel filter was used as a partition between the bath and the accumulator to which high purity argon was fed in. #150 zirconia was employed as the fluidized cooling medium in consideration of density, heat transfer rate, and high temperature stability. The temperature was measured at along the center line of the solidifying bar sample, René 80, and the location of the liquidus-solidus interfaces, the temperature gradient, G, and local solidification rate, R, were estimated by making use of polymonially approximated temperature fields during DS.

The FBQ method was applied to in-situ growth of $\gamma/\gamma'-\alpha$ eutectic composites with 1580°C hot zone temperature and at 2.6 cm/h DS rate. The master heat of the eutectic with the chemical composition, 32.3 Mo - 5.7 A.1 - Bal.Ni (wt %), was prepared by the vacuum melting from high purity stocks. Conventional optical microscopy and transmission electron microscopy(TEM) were used to investigate the structure of the as-grown, solutionized(SHT), and aged composites. Crystallographic identification of the microphases in the eutectic was made by the selected area diffraction method. The mechanical property of the composite and the influence of the heat treatments were studied through the creep rupture and micro-hardness testing.

3. RESULT AND DISCUSSION

Of the thermal analyses obtained from the temperature distribution in HRS, FBQ, and LMC process conducted for the René 80 bar sample, the change of G, and R values at the interface were calculated as a function of the solidification time. A representative G value change at the solidus interface is illustrated in Fig. 1. G by FBQ and LMC is about 10 times as much as that of HRS, and it increases gradually lowing the mold. G/R ratio is also 10 times as much on the average, demonstrating a great improvement in cooling effect. It is found however that there is little difference between FBQ and LMC. In the FBQ process, the fluidized bed temperature was measured. At a gas flow rate of less than 4ℓ/min where zirconia particles remained unfluidized, the bath temperature increased as the casting was inserted. When the bath was fluidized, its temperature was held at 100 to 120°C uniformly, and proved to be kept on the overall bath.

The TEM observation for a transverse section of a plane front growth ingot of $\gamma/\gamma'-\alpha$ eutectic identified three major phases in the microstructure as shown in Photo. 1. Mo fibers, α phase, of a square or rectangular cross section were encircled by the ordered γ' phase, and the remaining space was filled primarily with γ phase. In the longitudinal direction, Mo fibers were observed to maintain the homogeneous width without termination or branching unless the steady state growth was disturbed by local bandings. The crystallographic relationship between γ' and Mo fibers was found in accordance with the result previously reported, namely for the lattice orientation, [110]α//[100]γ', (100)α//(100)γ', and for the interface orientation, mainly (110)α//(100)γ' but partially (100)α//(110)γ'. A selected area diffraction analysis of the multi-phase region, primarily γ phase, suggested that the region consists of γ'($L1_2$-ordered), Ni_3Mo(DO_{22}), Ni_2Mo (body centered orthohombic), and γ (fcc) matrix phases: The characteristic microstructure of the as-grown composite is ascribed to the eutectic solidification followed by the peritectoid reaction and subsequent precipitation of the coherent particles in the γ matrix during cooling. When the eutectic was solutionized at 1260°C, the γ' phase, surrounding Mo fibers in the as-grown stage, markedly diminished, and resultant structure was made of Mo fibers embedded in γ' particle dispersed γ matrix as illustrated in Photo. 2. If the solutionized sample was reheated at 1260°C, and subsequently furnace cooled stepwisely holding 1h at every 100°C interval,

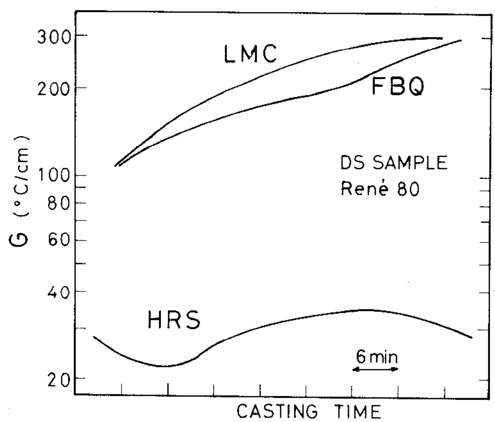

Fig. 1 Relative Magnitude of Thermal Gradient G for HRS, FBQ and LMC

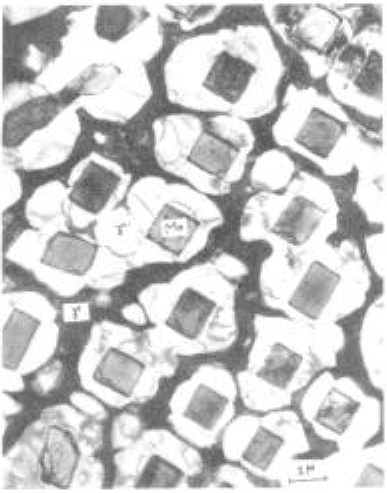

Photo. 1 TEM Micrograph of As-grown γ/γ'-α

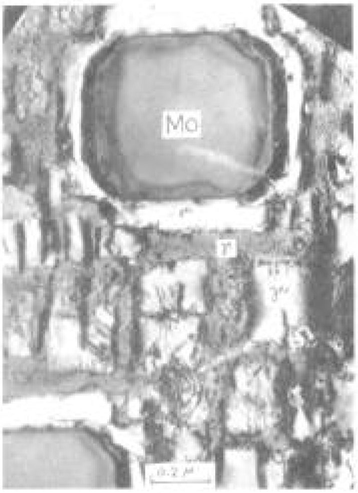

Photo. 2 γ/γ'-α Quenched from 1260°C

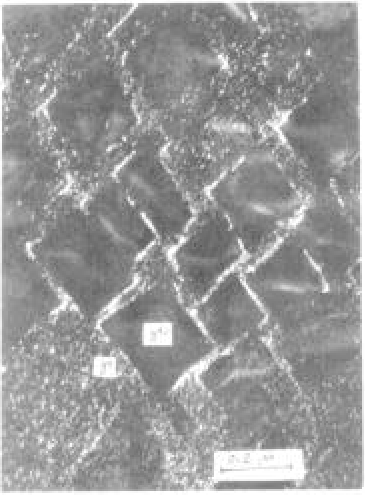

Photo. 3 Ni_2Mo Particles in γ/γ'-α Quenched and Aged at 700°C x 10h

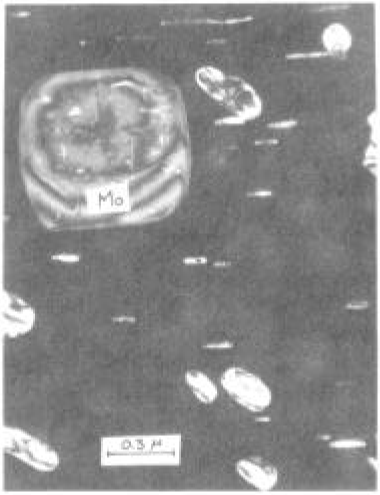

Photo. 4 Mo Platelets in γ/γ'-α Quench and Aged at 900°C x 10h

the microstructure identical to that of the as-grown stage as seen in Photo. 1 was obtained, and thus the existence of the peritectoid was evident.

Aging treatment of the solutionized samples induced two different precipitates in γ phase depending upon the aging temperature and time. At temperatures below 830°C, a fine coherent metastable phase of orthorhombic Ni_2Mo (DO_{22}) was formed. By aging above 830°C, Mo pletelets precipitated in the γ matrix at the expense of the metastable Ni_2Mo. Photos. 3 and 4 show TEM dark field micrographs of Ni_2Mo particles at 700°C aging, and Mo platelets precipitates at 900°C aging, respectively.

The result of the creep rupture test is summarized in Table 1. As-grown samples with the full plane growth condition showed excellent creep performance while the samples with the casting defects of cell or local banding structures failed at relatively short time. The significant reduction of the rupture strength was also noted for the samples to which SHT or SHT+AGE treatments were given. This was presumably caused by the strong internal strain fields associated with the dense dislocations observed in γ/γ' matrix of the heat treated composites as seen in Photo. 2. The microhardness of the composites solutionized at various temperatures between 1100°C and 1290°C indicated marked hardening for the samples quenched from the temperatures above 1215°C, and simultaneous TEM study identified the peritectoid reaction at this temperature. The influence of the aging temperatures on the hardness is currently investigated. Appreciable change in the hardness has been observed at the aging conditions whereat Ni_2Mo precipitations were confirmed by TEM.

4. CONCLUSION

In the experimental evaluation of FBQ directional solidification parameters, it was found that FBQ offers a thermal gradient about 10 times as much as HRS, and is almost equivalent with LMC so far as the cooling capability is concerned, suggesting FBQ to be a highly prospective engineering technique for DS.

The TEM observation have demonstrated that as-grown composites of 32.3 Mo - 5.7 Al - Bal.Ni (wt %) alloy consists of a well aligned Mo fibers surrounded by the ordered γ' phase and γ phase which is a multi-phase mixture including γ', coherent Ni_3Mo and Ni_2Mo. The as-grown structure is formed by the eutectic and the peritectoid reaction followed by the precipitation in the γ phase during cooling below the peritectoid temperature. The microstructure and hardness can be altered markedly by appropriate thermal treatments. Quenching above the peritectoid temperature results in the microstructure consisting of Mo fibers surrounded by a fine mixture of γ and γ',

Table 1. Result of Creep Rupture Test

C. CELL, E. EUTECTIC, B. BANDING

	HEAT TREATMENT	MICRO-STRUCTURE	STRESS (kg/mm^2)	TEMP. (°C)	RUPTURE TIME (h)	ELONGATION (%)
1	As-grown	C.	24.5	982	0.5	—
2	As-grown	Partly C.	79.1	760	48.2	6.0 *1
3	As-grown	E.	79.1	760	> 54.8	—
4	As-grown	E. (B)	24.5	982	17.1	1.8 *2
5	As-grown	E.	24.5	982	> 50.1	—
6	As-grown	E.	24.5	982	110.8	6.6
7	SHT	E. *3	24.5	982	21.7	13.2
8	SHT+AGE	E. *4	24.5	982	30.8	21.1

REMARKS: *1 Ruptured at Cell, *2 Ruptured at Banding
*3 SHT (1260°C x 2h), *4 AGE (845°C x 16h)

however, the high temperature strength is reduced by the quenching treatment because of the dense dislocations existing in this state. When a quenched sample is aged below 830°C, a metastable coherent Ni_2Mo precipitates in γ phase, and causes appreciable hardening. Aging above 830°C induces Mo platelets precipitation with different orientation relationship from the Mo fibers.

5. REFERENCES

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