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Phase Transformation Studies at the Solid/Liquid Interface by Directional Solidification of Alloy 718

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The phase transformation temperatures are especially important in alloy 718 because it requires careful heat-treatment cycles. The DTA method is generally used to estimate the characteristic temperatures of phase transformations. However, DTA is an indirect method to confirm phase transformations, and there are always discrepancies in analyzing the DTA thermograms. In this study, the directional solidification and quenching technique has been applied to estimate the phase transformation temperatures near the solid/liquid interface temperatures by measuring temperatures directly during directional solidification. These phase transformation temperatures were confirmed in the quenched solid/liquid interface. Solidification behavior at the solid/liquid interface with solidification rate and temperature gradient were also discussed.

Keywords : phase transformation, DTA, directional solidification, dendrite, interdendritic region

I. INTRODUCTION

Alloy 718 is widely used in gas turbine components due to its excellent high temperature mechanical properties, corrosion resistance and weldability [1,2]. However, the cast 718 alloy inevitably evolves severe segregation of Nb content which forms the major strengthening phase of "during solidification. The segregation causes not only microstructural instability but also the formation of the Laves phase and porosity, which are harmful to mechanical properties and weldability; therefore, homogenization treatment is required for the cast 718 alloy. Solidification and segregation behaviors are fundamental subjects to be studied, but most research has been focused on the relationship between microstructure and mechanical behavior of the forged 718 alloy. Solidification and phase transformation studies have been mainly performed by DTA (Differential temperature analysis). The phase transformation temperatures are especially important in alloy 718 because it requires careful heat-treatment cycles, such as homogenization, solution treatment, and several steps of aging [3]. DTA results always include problems of analyzing the phase transformation temperatures in the thermograms as there often occurs some discrepancies in the same thermogram [4-8]. In order to eliminate these discrepancies, a directional solidification with quenching technique has been applied to estimate phase transformation temperatures by measuring temperatures directly during directional solidification. These phase transformation temperatures were confirmed in the quenched solid/liquid interface. Solidification microstructure and solid/liquid interface morphology were also examined with solidification rate and temperature gradient.

2. EXPERIMENTALS

Alloy 718 was directionally solidified in 8 mm-OD (OD: outer diameter) by 5 mm-ID (ID: inner diameter) recrystallized Al_2O_3 tubes in the modified Bridgeman type furnace that allowed quenching of the solid/liquid interface [9]. The master ingot was supplied from Cannon-Muskegon and its nominal composition is shown in Table 1. In order to prepare a sample to fit into the alumina tube, 4.8 mm dia. rods were prepared by electric discharge machining. A Super-Kanthal heated furnace fitted with a water-cooled Cu toroid at its bottom end was passed upward at controlled rates around the alumina tube filled with the alloy under an Ar atmosphere. After about half of the original liquid had been solidified the alumina tube was dropped into a quench bath of stagnant water, thereby preserving the solid microstructure formed at

Table 1. Chemical composition of cast alloy 718

Element	C	Mn	Si	P	S	Cr	Mo	Nb+Ta	Ti	Al	Fe	Ni
Wt.%	0.08	0.35	0.35	0.015	0.015	19	3.0	5.1	0.90	0.60	18	Bal

the solid/liquid interface.

The emphasis of these experiments was placed on accurately measuring the temperature gradients during solidification. The temperature measurements were made with Pt-6% Rh/Pt-36%Rh, B-type thermocouple wire. The thermocouple consisted of double hole Al_2O_3 containing 0.13 mm dia. wires surrounded by a 1.5 mm OD Al_2O_3 tube and it was calibrated by solidifying pure Cu and Ni samples. The assembled thermocouple was inserted axially from the top of the molten metal to just above the solid/liquid interface and the temperatures were recorded as the furnace and cold finger were moved up the axis of the molten metal causing solidification of the metal around the thermocouple.

DTA Analysis was performed using a computer controlled Perkin-Elmer DTA 1700 analyser under pure argon gas flow. The weight of the test specimen was 70-100 mg approximately. The furnace temperature was calibrated by the Pt/Pt13%Pt R-type thermocouple. The heating/cooling rate was 5°C/min.

3. RESULTS AND DISCUSSION

3.1. Solidification microstructure

Directional solidifications were carried out at the solidification rates of 0.5-100 μm/s in alloy 718. The solidification microstructure and solid/liquid interface morphologies are summarized in Table 2. The dendritic solid/liquid interface changed to a cellular interface with decreasing G/V values (G: temperature gradient, V: solidification rate), as shown in Fig. 1. Interface stability is highly expected because of the constitutional supercooling [10]. Well developed dendrites were found at relatively high solidification rates of 25-100 um/s. Fig. 2 displays micrographs of the transverse section which is perpendicular to the solidified direction. The microstructures are characterized by EPMA (Electron probe microanalysis) in SEM, as shown in Fig. 3. The Laves phase was found in the interdendritic region where the final freezing occurred; however, MC carbides existed in not only the dendritic region but also in the interdendritic region. The EPMA analysis showed that the carbides were mainly NbC with some of TiC. The carbides were found from near the center of dendrite to the edge of dendrite, as shown in Fig. 3(a). It indicates that the carbide forms near the dendrite tip region. The carbide formation temperature will be discussed in the next section. The delta phase found around the Laves phase where Nb segregated, as shown in Fig. 3(b). The delta phase appears to form around the Laves phase at the solid/liquid interface of

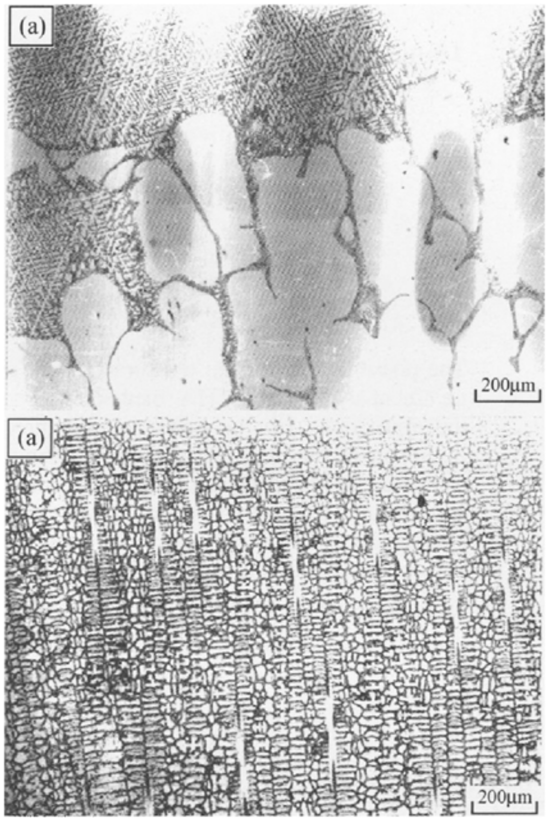

Fig. 1. Quenched solid/liquid interface mophologies. (a) 0.5 μm/s, and (b) 50 μm/s.

Table 2. Solidification microstructures with solidification rates

V(μm/s)	G*(C/cm)	G/V(× 10^5°C sec/cm^2)	Dendrite/Cell length (mm)	Dendrite/Cell spacing (μm)	Interface morphology
0.5	78	15.6	-	-	Cellular
5	76	1.5	-	-	Cell./Dend.
25	78	0.3	16	275	Dendritic
50	75	0.15	21	235	Dendritic
100	74	0.07	21	213	Dendritic

*G is the gradient in mushy zone

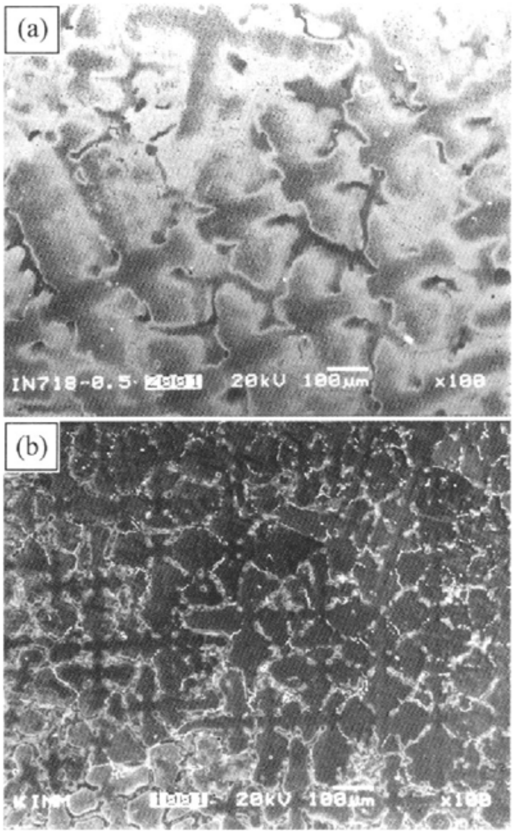

Fig. 2. Cross sections of directionally solidified rods. (a) 0.5 µm/s, and (b) 50 µm/s.

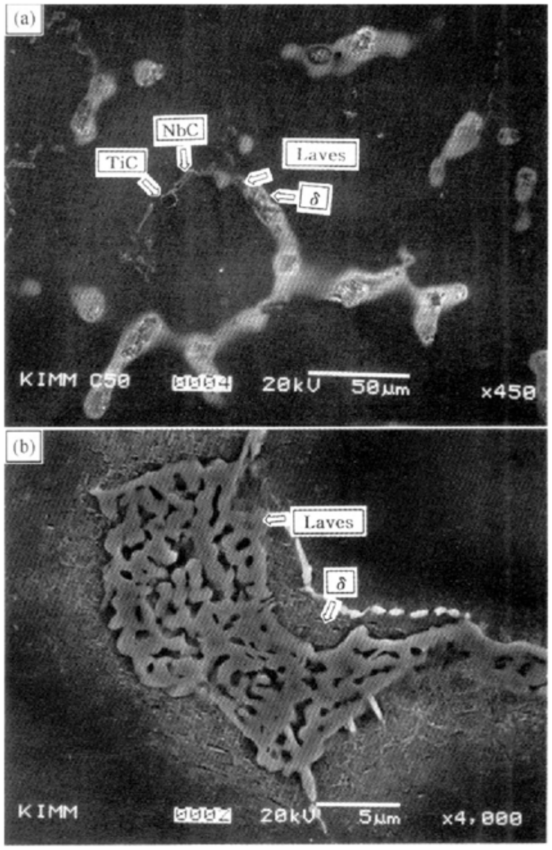

Fig. 3. Microstructure of directionally solidified 718 alloy. (a) High magnification figure of Fig. 2(b) and (b) High magnification figure of Fig. 3(a), where the delta precipitates are shown around the Laves phase.

is known to be related with the content of Nb, and occurs at and below 1010°C [3]. We expect the precipitated delta phase at the solid/liquid interface in the interdendritic region to form during the quenching process to cool down from the solid/liquid interface temperature to room temperature.

The primary dendrite spacing decreased with solidification rates. The primary dendrite spacing is predicted by temperature gradient (G) and solidification rate (V) according to [11].

$$d=K\,V^{-0.25}G^{-0.5} \qquad (1)$$

where K is constant. The dendrite spacing in this experiment was aligned well with this equation and it is compared with the other data [12], as shown in Fig. 4.

The microsegregation that occurred during solidification is clearly illustrated by analyzing the composition variation between two dendrite cores, as shown in Fig. 5. The carbide and Laves phase forming elements, such as Nb, Mo, Ti, segregated in the interdendritic region. It is shown that Nb is heavily segregated in the interdendritic region and the segregated Nb plays a major role in forming NbC, Laves, and delta precipitation.

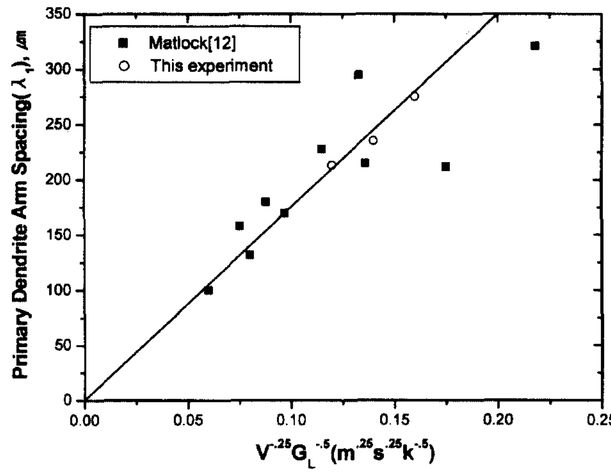

Fig. 4. Dependence of the primary dendrite arm spacing on G(gradient) and V (solidification rate).

3.2. Phase transformation temperature

The phase transformation temperatures are usually characterized by DTA. The equilibrium temperature can be obtained when the heating or cooling rate approaches zero theoretically. However, it is practically impossible to make the heating or cooling rate zero. The results are very sensitive to heating

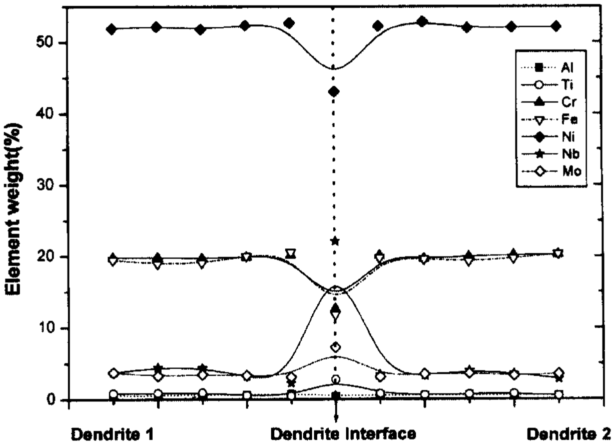

Fig. 5. Segregation analysis between dendrite and interdendrite in the 50 μm/s directionally solidified rod.

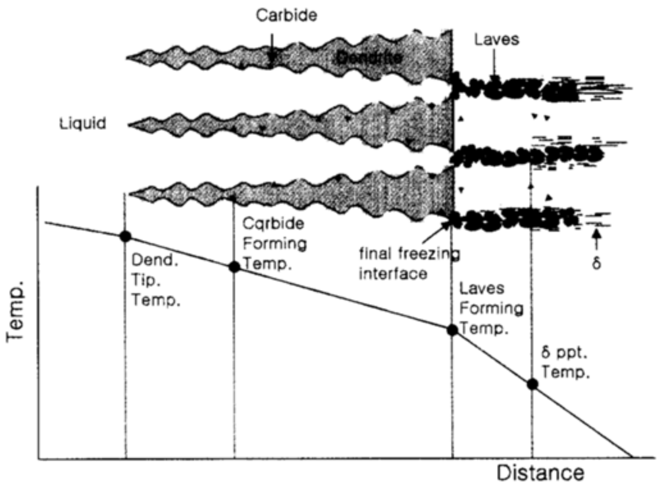

Fig. 6. Schematics of comparison between growing dendrite and temperature gradient at the solid/liquid interface during directional solidification.

or cooling rates, for instance, slow heating rates result in weak characteristic peaks and fast rates decrease the resolution of the peaks. The phase transformation temperatures of the 718 alloy have been investigated by a number of investigators [4-8]. The recent data are summarized in Table 3, and there is some discrepancy among the data. These deviations might be caused by the differences in the measuring methods, cooling or heating rates, and sample conditions or composition. Especially, the temperatures of phase transformation may be vary because of the different heating or cooling rates.

The directional solidification by quenching technique was applied to estimate the characteristic temperatures near the liquid/solid transformation region. With direct measurement of the liquid, liquid/solid interface, solid phase temperatures during directional solidification process, liquidus and final freezing temperature were estimated. At the same time, these temperatures were verified by DTA. Fig. 6 shows the schematic diagram of growing dendrite and temperature profile during directional solidification. The dendrites of the γ phase form first, and the interdendritic melt is gradually enriched by solute elements to form the carbides and Laves phase. The formation temperature could be estimated by comparing the

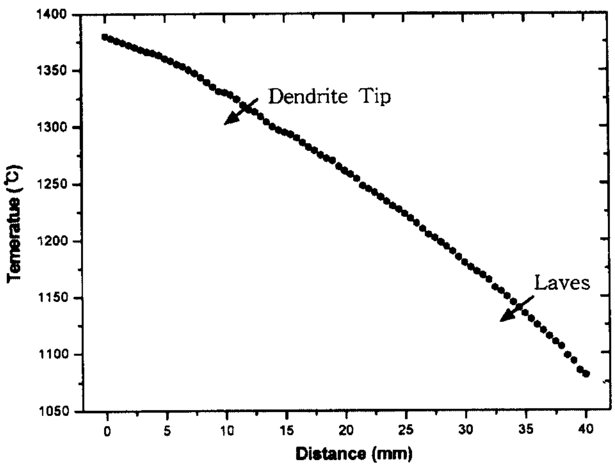

Fig. 7. Temperature profile in solid and liquid during directional solidification at 50 μm/s.

quenched interface and the temperature profile during directional solidification. Fig. 7 shows the temperature profile at the solidification rate of 50 μm/s, where the temperature of

Table 3. Characteristic temperatures of phase transformation in Alloy 718

Temp. (°C) Data	Liquidus		NbC		Laves		Delta	
	Cooling	Heating	Cooling	Heating	Cooling	Heating	Cooling	Heating
Dahotre [4] (Cast)	1319	1351	1249	1259	1160	1180	1132	1136
Dahotre [4] (Wrought)	1323	1354	1262	1282	1152		1098	
Cieslak [5]		1351	1257	1230	1185			
Ballantyne [6]	1340		1270		1140-1173			
Cao [7]	1315	1345	1262	1267-1305	1162	1167		
Murata [8]	1359		1289		1160		1145	
This study by DTA	1310	1343		1280	1162	1169		
This study by D/S	1330		1295		1165		-	

liquid, solid/liquid interface, and solid were directly measured during directional solidification. The break points at the dendrite tip and final freezing position were found in the temperature profile. It is known that the well developed dendrite tip temperature is close to the liquidus in the directional solidification condition [10]. The dendrite tip temperature is expected to be 1330°C in the temperature profile. Another break point was found at 1165°C, which is expected to be the final freezing temperature and the Laves phase formation temperature because it is known that solidification ends up with formation of the Laves phase in 718. The second break point is not so clear because the temperature profile in the mushy zone is not linear. However, the formation temperature of the Laves phase could be confirmed by DTA result and the length of the dendrite. The second break temperature of 1165 °C in Fig. 7 agreed with the Laves formation temperature of 1162-1169°C by DTA. On the other hand, the final freezing temperature might be deduced by the length of the dendrite, which was measured at the solid/liquid interface, and the temperature gradient. The final freezing temperature could be estimated to be 1172.5°C in the following simple equation [10], if one consider that the dendrite length is 21 mm, temperature gradient is 75°C/cm in the mushy zone, and dendrite tip temperature is 1330°C.

$$G=(T_{tip}-T_{base})/L \tag{2}$$

where L is the length of the dendrite. The final freezing temperature estimated by the length of the dendrite shows some deviation from the results by temperature profile and DTA. There might be some error in estimating the final freezing temperature because the solidification interface in the interdendritic region is not so clear. The dendrite length was 21 mm, which was measured in the directionally solidified sample and 22 mm in the temperature profile of Fig. 7.

The carbide formation temperature was estimated as 1292.5°C because the first trapped carbide was found 4 mm below the dendrite tip, where the gradient was 75°C/cm. It was difficult to estimate the formation temperature of the delta phase in the directionally solidified sample. This phase was found around the Laves phase at the solid/liquid interface is known to be precipitates from an Nb enriched solid in the temperature range of 871-1010°C [3]. There must be a delta phase free region at the solid/liquid interface; however, the phase is expected to precipitate during the quenching process because it is formed with the Laves phase at the quenched solid/liquid interface. Therefore, the temperature at which the delta phase begins to precipitate could not be estimated in this study. A faster quenching method, such as using GaIn liquid metal quench media, might make it possible to delay the delta phase precipitation during the quenching process.

4. CONCLUSIONS

Directional solidification by the quenching technique has been applied to estimate phase transformation temperatures and to examine solidification behavior of the In718 alloy near the solid/liquid interface temperature.

(1) Dendrite spacing was governed by the equation $l=K\,V^{-0.25}\,G^{-0.5}$.

(2) Heavy segregation of Nb as well as other elements Cr, Mo, and Ti, which form carbides and the Laves phase, was found in the interdendritic region.

(3) By measuring temperatures directly during directional solidification from liquid to solid, the liquidus, formation temperatures of NbC and Laves were estimated. Those characteristic temperatures made by the directional solidification method were between the estimated values from the cooling and heating of previous DTA results. It was interesting to find that those were closer to the values from heating than to those from the cooling of DTA.

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