

DESIGN OF SOLUTIONIZING HEAT TREATMENTS FOR AN EXPERIMENTAL SINGLE CRYSTAL SUPERALLOY

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Abstract

The heat treatment response between 1170°C and 1320°C of an experimental single crystal Ni base superalloy is presented. The temperature range for solutionizing the alloy is determined by combinations of holding time and temperature. The effects of long isothermal holding within and beyond this solutionizing temperature range are studied. Heat treatment steps below the γ' -solvus temperature stabilize the eutectic phase, while steps above the solvus temperature improve the homogenization and reduce eutectic phase fraction. However, prolonged holding above the solvus temperature indicates up-hill diffusion at the eutectic region leading to incipient melting. A new solutionizing heat treatment approach with continuous heating between solvus and solidus is proposed

Introduction

As cast microstructures of Ni-base superalloys are highly heterogeneous due to coring during solidification [1,2]. To increase the high temperature capabilities, modern single crystal (SX) superalloys are alloyed with the refractory elements, W, Ta, and Re [3]. As a consequence, the microsegregation of these alloys is being more pronounced leading to higher eutectic fractions [4] and chemical instabilities [5]. Hence, heat treatment of modern SX alloys is becoming an increasingly challenging, time consuming and expensive process [6]. For the most part, the specific heat treatment processes of commercial as-cast superalloys are treated as empirical proprietary knowledge by superalloy designers and producers.

To promote chemical homogeneity and eliminate coring, as well as achieve microstructural uniformity, as-cast alloys undergo solution heat treatment. To obtain the desired uniform γ/γ' microstructure, the solution heat treatment is followed by γ' precipitation aging. The solution treatment should dissolve the γ' phase (both within the dendrites and interdendritic eutectic) into the γ matrix by either isothermal or stepwise heat treatment, between the solvus and solidus temperatures, which is commonly referred to as 'solutionizing window' [7], followed by quenching. The aging treatment involves holding the alloy at sub-solvus temperatures to obtain fine and uniform γ' precipitates within the γ matrix. To promote a bimodal distribution of γ' phase, aging often involves high temperature primary aging, followed by lower temperature secondary aging.

Despite the above understanding, it is challenging to design solution heat treatments for new superalloys because of the following reasons.

1) Differential Scanning Calorimetry (DSC) is generally employed to obtain phase transformation temperatures. However, it is difficult to identify solvus and solidus temperatures of an as-cast superalloy from the DSC profiles because of the interference of these transformations due to chemical heterogeneity [8].

2) It may be possible to resolve solidus and solvus peaks in a DSC thermograph following some degree of homogenization. However, these phase transformation temperatures not only vary with the heating or cooling rate but also with the degree of homogenization [9].

3) A highly segregated as-cast microstructure may have a solidus temperature lower than the solvus temperature. Under such a circumstance, a complete solutionizing heat treatment is considered impossible [10]. It is not uncommon that segregation of trace elements P, S, Si, B and Hf promote incipient melting in the interdendritic region at very low temperatures [11-13].

4) The complete dissolution of eutectic γ' is difficult because of its coarse blocky morphology; a low surface area per volume makes it dissolve very slowly into γ matrix [14].

The purpose of this research is to propose an efficient procedure for designing solutionizing-homogenizing heat treatments for heavily alloyed SX superalloys. The application of the proposed procedure is illustrated utilizing a newly designed experimental single crystal superalloy with significant refractory content. Stepwise heat treatments followed by microstructural analysis are employed to identify the solutionizing window. The effects of holding time within and outside this solutionizing window are discussed using EPMA (Electron Probe Microanalysis) and phase fraction analysis. Based on the results and analyses, a new solutionizing-homogenizing heat treatment approach is proposed.

Experimental Material and Methods

An experimental nickel base superalloy based on PWA 1480, with the nominal composition listed in Table I was used for the present research. The alloy was cast with a withdrawal rate of 5.65×10^{-5} m/s (8.0 in/hr) into SX bars of 16 mm diameter and 200mm length in a Bridgman furnace using PWA 1484 SX starter seeds [4]. The SX bars with the longitudinal axes along $\langle 001 \rangle$ were sectioned in the transverse direction to obtain discs of approximately 6 mm thickness.

Table I: Nominal composition (wt %) of experimental superalloy

Ta	W	Re	Co	Mo	Cr	Ti	Al	Ni
12	2	2	5	2	7.5	1	5	Balance

Table II: Heat treatment experiments

Expt. No.	Heat Treatment
1	1170°C/15h + 1250°C/2h + 1280°C/2h + 1300°C/2h + 1310°C/2h
2	1170°C/15h + 1250°C/2h + 1280°C/5h + 1300°C/2h + 1310°C/2h + 1315°C/2h + 1320°C/2h
3	1170°C/2h + 1250°C/2h + 1280°C/15h + 1300°C/2h + 1310°C/2h + 1320°C/2h
4	1170°C/2h + 1250°C/2h + 1280°C/5h + 1300°C/2h + 1310°C/5h + 1315°C/15h
5	1170°C/2h + 1250°C/2h + 1280°C/5h + 1300°C/5h + 1310°C/15h
6	1280°C/2h + 1295°C/2h + 1305°C/2h + 1310°C/2h
7	1280°C/2h + 1295°C/2h + 1305°C/10h
8	1280°C/2h + 1295°C/2h + 1305°C/20h

A box type radiant furnace with accurate temperature control of $\pm 1^\circ\text{C}$ was used for heat treatments. An additional thermocouple with its bead in contact with the specimen was used to confirm the temperature measurement. Table II lists stepwise heat treatment experiments in which each temperature step follows from the previous temperature. The temperature range of 1170 to 1320°C was chosen based on the DSC thermograph of the alloy obtained by Kearsey et al. [4]. To track the microstructural evolution during the stepwise heat treatments specimens were quenched in water after each step. All the experiments were performed in an air atmosphere. To avoid the interference of surface oxidation in the analysis, the specimen discs were sectioned transversely at the centre of 6mm thickness and the unoxidized surfaces were used for metallographic examination. Specimens were prepared for optical microscopy by grinding to 1 μm finish using standard metallographic procedures, followed by etching in a solution of 0.3 g molybdic acid, 10 ml HNO_3 , 10 ml HCl and 15 ml H_2O .

Eutectic Phase Fraction Analysis

To track the degree of solutionizing, eutectic phase fraction analysis was carried out using the image analysis software, Image Tool™. A representative low magnification optical micrograph of the alloy obtained after each heat treatment step was converted into 16-bit grayscale format. After adjusting the brightness and contrast to delineate the dendrite and eutectic region, the grayscale format was converted into black and white threshold image using an Image Tool™ subroutine. Residual pixels corresponding to the eutectic region present in the black and white images were removed manually using a pixel editing tool. Finally, areas corresponding to dendrite and eutectic regions were obtained by counting the pixels from the black and white regions.

Electron Probe Microanalysis (EPMA)

It has been established that a sharp compositional gradient exists in the as-cast alloy from the interdendritic eutectic to dendrite core region along [011] type direction [4]. Hence, studying the variation of this composition profile after each heat treatment step gives an estimate of degree of homogenization and hence the effectiveness of the heat treatment step. To study the variation of composition profiles, a series of spot chemical analysis was performed with intervals of about 25 μm along a line from the eutectic region to the dendrite core.

A Camebax Electron Microprobe with Wavelength Dispersive X-Ray (WDX) spectrometers was used for the analysis. Selected specimens ground to 1 μm finish by standard metallographic techniques were used for the analysis. The electron probe with accelerating voltage, 20 kV and beam current, 35 nA was used for the analysis over 5 μm x 5 μm raster with total counting time of 60 s for each element. A well characterized suite of pure metals and minerals was used for calibration. The matrix correction software, Cameca PAP, was used for converting X-ray data into elemental weight percent. The limits of background noise levels were carefully determined by running wavelength scans on either side of the peak position of each analyzed element. As a result, highly accurate quantitative results were obtained including elements added in small amounts (Re, Ti, W).

Results and Discussion

Microstructural Analysis

The as-cast microstructure of the alloy is shown in Figure 1. The dendrite core microstructure is fine and uniform, while interdendritic region contains a cellular eutectic phase.

Figure 2 shows the microstructural developments during the stepwise heat treatment identified as experiment 1 in Table 2. The first microstructural change occurs during the 1250°C step as the interdendritic region around the eutectic phase becomes denuded of fine γ' precipitates, indicating that solutioning begins at the interdendritic region. Kearsey et al. [2,4] studied and confirmed this using scanning electron microscopy. However, during the higher temperature steps, this γ' denuded region does not spread to dendrites. Instead, dendritic solutioning begins at the core region during the 1280°C step (Figure 2b) followed by complete solutioning of dendrites during the 1300°C step leaving behind undissolved blocky eutectic at the interdendritic region (Figure 2c). During the 1310°C step, incipient melting occurs in the interdendritic region adjacent to the residual eutectic (Figure 2d). From these observations, it may be approximated that γ' -solvus temperature is 1290°C and the solidus is 1310°C and hence, the solutioning window is approximately 1290°C to 1310°C.

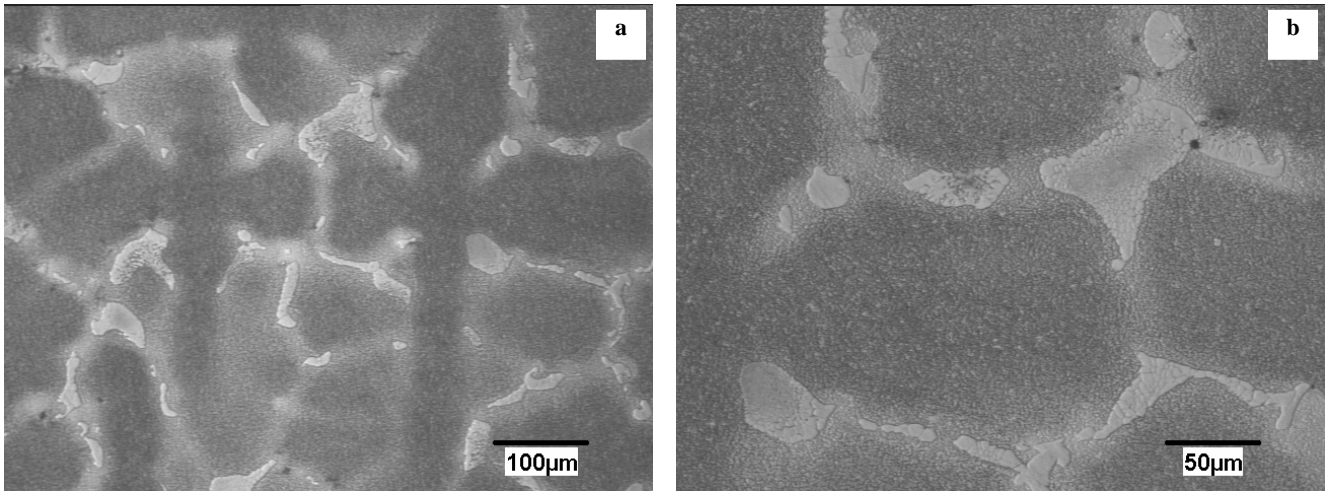


Figure 1: As-cast microstructure of the alloy showing: a) dendritic structure, b) cellular eutectic phase at the interdendritic region

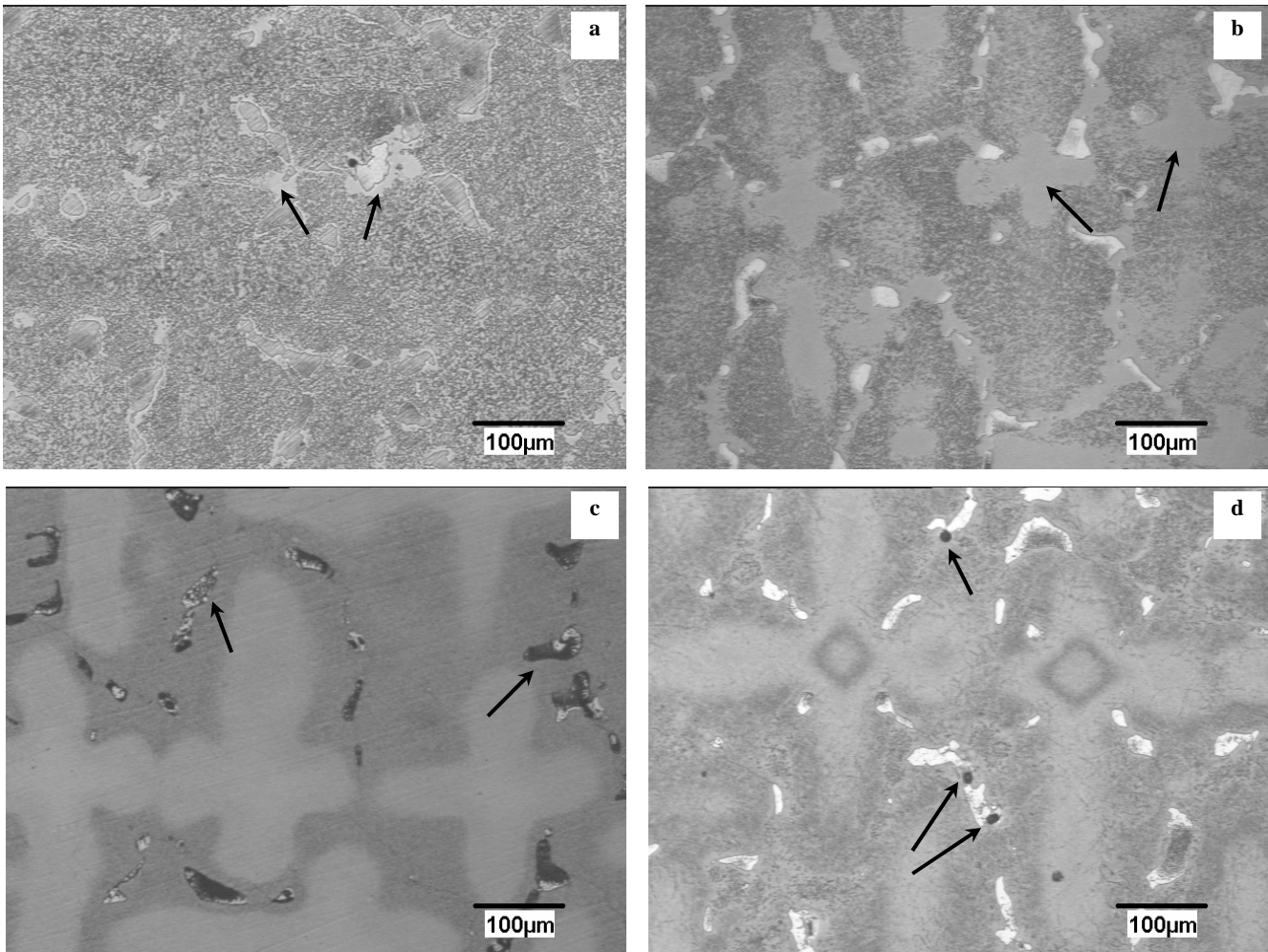


Figure 2: Microstructure of the alloy after: a) 1170° C/15h+1250° C /2h showing denuded region around the eutectic, b) same as a) +1280° C/2h showing solutioning at dendrite cores, c) same as b)+1300° C /2h showing the residual eutectic phase between fully solutionized dendrites, d) same as c) +1310° C /2h showing incipient melting at the interdendritic region

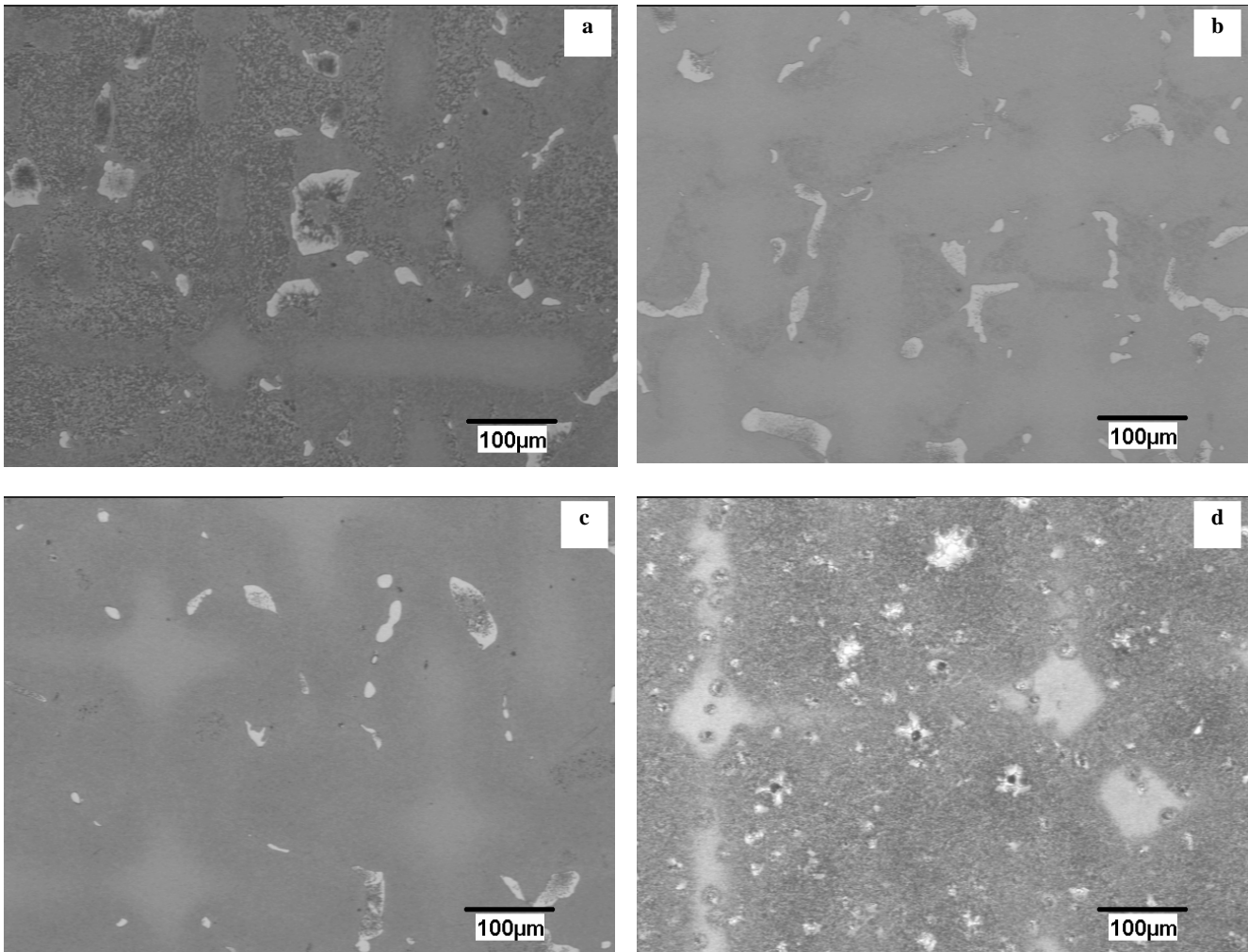


Figure 3: Microstructures of the alloy after: a) $1170^{\circ}\text{C} / 15\text{ h} + 1250^{\circ}\text{C} / 2\text{ h} + 1280^{\circ}\text{C} / 5\text{ h} + 1300^{\circ}\text{C} / 2\text{ h}$, b) same as a) $+ 1310^{\circ}\text{C} / 2\text{ h}$ showing no sign of incipient melting, c) same as b) $+ 1315^{\circ}\text{C} / 2\text{ h}$ showing reduced eutectic phase fraction, d) same as c) $+ 1320^{\circ}\text{C} / 2\text{ h}$ showing incipient melting.

The results of experiment 2 depict the effects of extended holding (5h) at a sub-solvus temperature of 1280°C . As observed from Figure 3, there is no sign of incipient melting of the alloy up to 1315°C . However, at 1320°C , the alloy shows considerable incipient melting at both interdendritic region and within the dendrites.

Figure 4 shows the occurrence of incipient melting after 1320°C step of experiment 3. A careful observation of microstructures of the alloy, before and after 1320°C step, confirms that the residual eutectic blocks at the interdendritic region undergo a eutectic reaction giving rise to cellular colonies.

From experiments 1, 2 and 3, it is clear that prolonged sub-solvus holding ($1280^{\circ}\text{C}/2\text{h}$, 5h , 15h) helps in raising solidus temperature slightly but not monotonically. Experiments 4 and 5 revealed incipient melting during prolonged holding at temperatures 1315°C , 1310°C respectively. Figure 5 illustrates how extended holding promotes incipient melting.

Figure 6 shows the results of experiments 6, 7 and 8 involving heat treatment steps within the solution heat treatment window ($1290\text{--}1310^{\circ}\text{C}$). A systematic reduction in the eutectic fraction is evident from the micrographs indicating that γ' solutioning is very effective within the window. However, a careful observation of figure 6d indicates onset of incipient melting and hence confirms the deleterious effect of prolonged holding.

From the microstructural observations, it may be summarized that γ' solutioning is effective within the solutioning window and that irrespective of the degree of homogenization or the thermal history of the alloy, extended holding in the vicinity of solidus temperature leads to incipient melting.

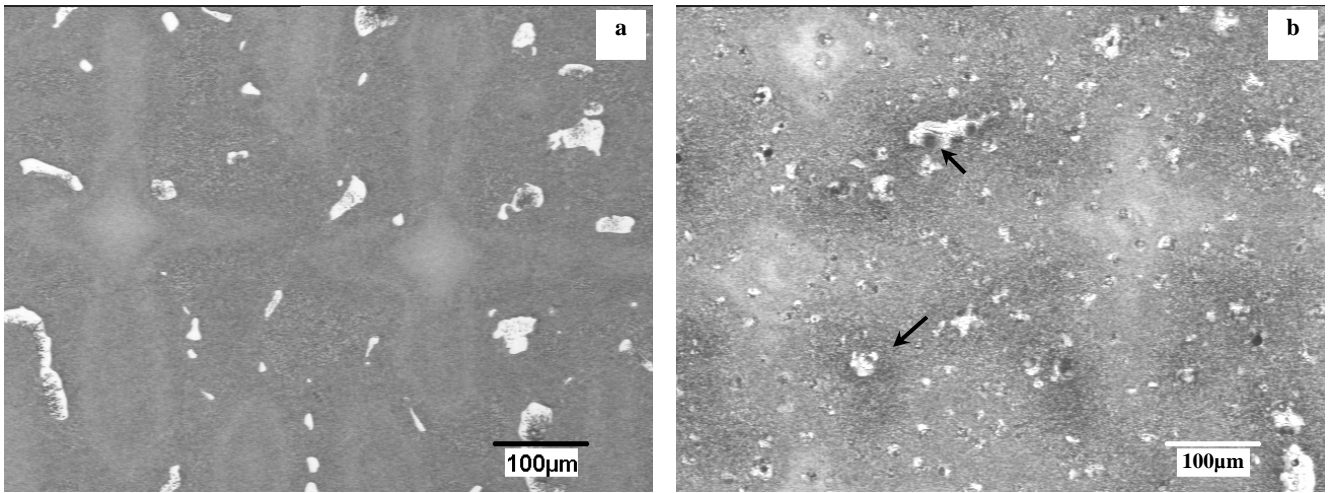


Figure 4: Microstructures of the alloy after: a) 1170° C /2 h +1250° C /2 h +1280° C /15 h +1300° C /2 h +1310° C /2h, b) same as a) +1320° C /2h showing a burst of incipient melting.

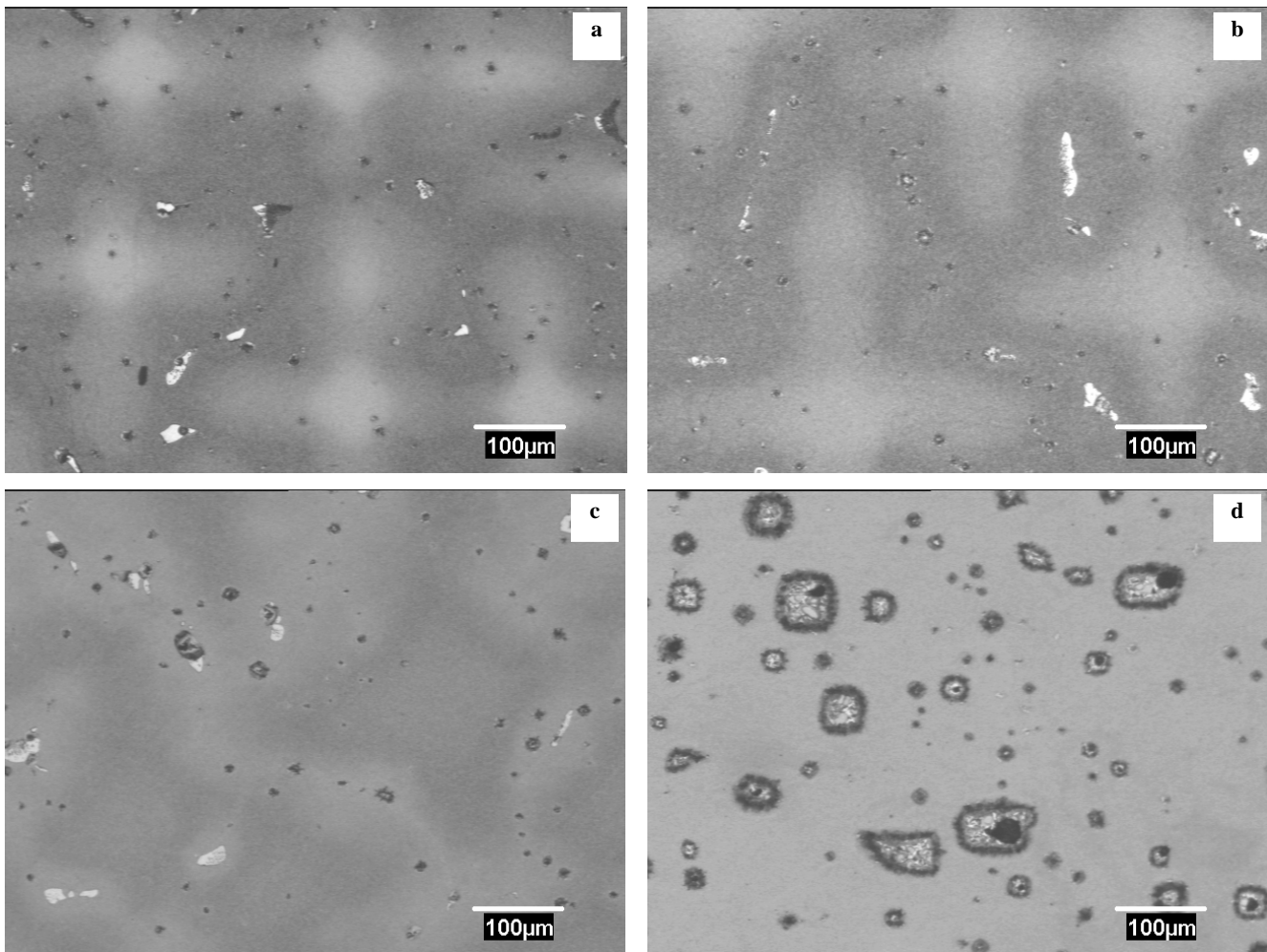


Figure 5: Microstructural change of the alloy after the step heat treatment 1170° C /2h+1250° C /2h+1280° C /5h+1300° C /5h followed by holding at 1310° C for: a)2h, b)5h, c)10h, d)15h showing a systematic development of incipient melting.

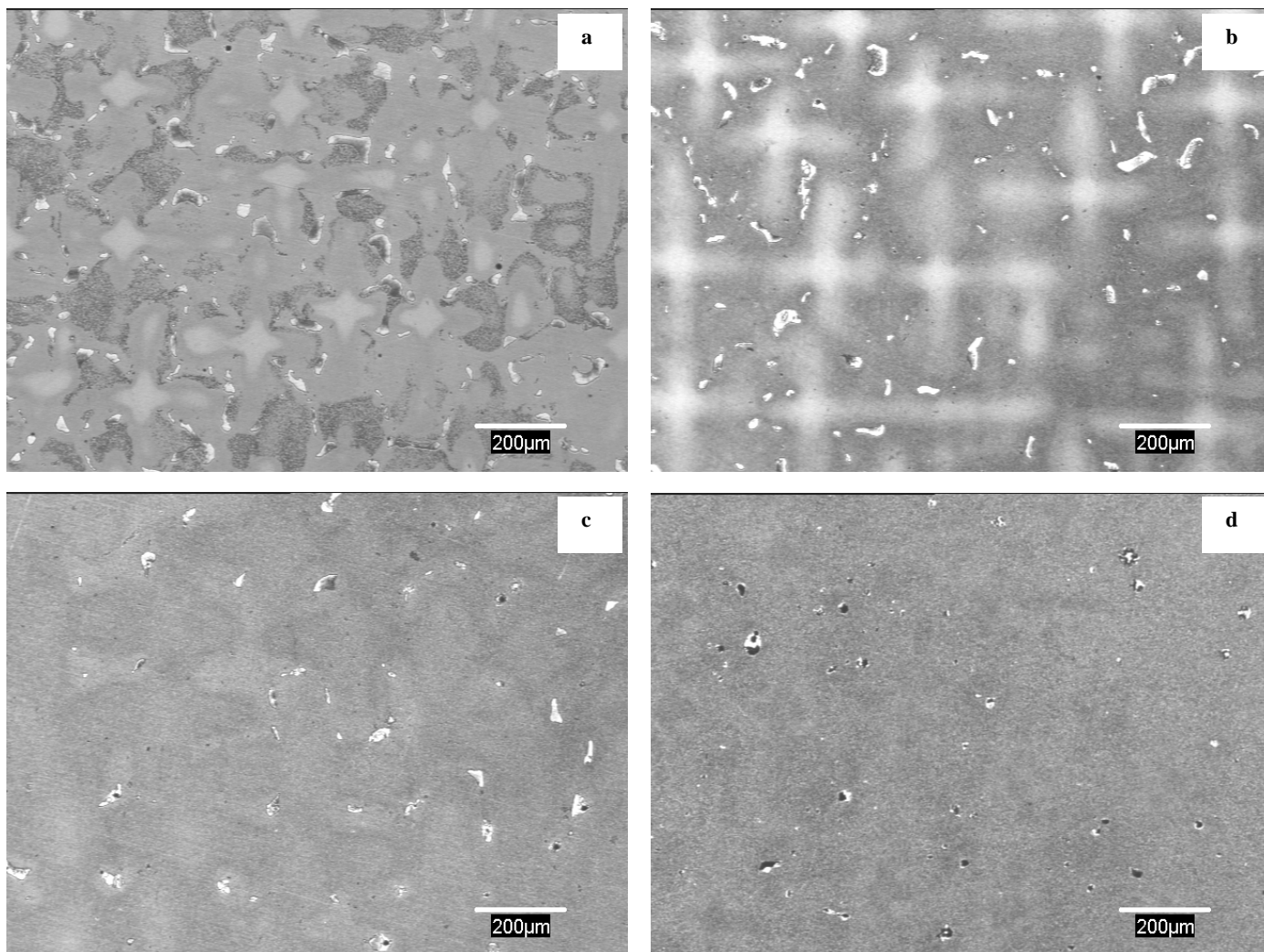


Figure 6: Microstructural change of the alloy after: a) 1280° C/2h+1295° C /2h, b) same as a) +1305° C /2h, c) same as a) +1305° C /10h, d) same as a) +1305° C /15h showing a systematic reduction of eutectic phase.

Table III: Eutectic phase fraction analysis

Expt. No	Heat Treatment Step	Eutectic Phase Fraction (%)
-	As-cast	6.99
1	1280°C/2h	3.29
2	1280°C/5h	3.38
3	1280°C/15h	4.23
6	1280°C/2h, 1295°C/2h, 1305°C/2h	4.76, 3.78, 2.33
7	1305°C/10h	1.26
8	1305°C/20h	0.91

Eutectic Phase Fraction Analysis

Eutectic phase fraction analyses performed on selected specimens are listed in Table III. Figure 7a and 7b illustrate the effects of extended holding on the solutioning of eutectic at a sub-solvus

temperature, 1280°C and a super-solvus temperature, 1305°C. Figure 7c shows the effect of increase in the holding temperature. Figure 7a indicates the unusual trend of increase in the eutectic fraction with increase in the holding time. This implies that an extended holding at 1280°C stabilizes the eutectic phase rather than dissolving it. However, extended holding above the solvus temperature shows a systematic reduction in the eutectic fraction, Figure 7b. The stepwise heat treatment with increase in the holding temperature also shows a monotonic reduction in the eutectic phase, Figure 7c. This indicates that a sub-solvus heat treatment has no beneficial effect on the dissolution of eutectic phase while a super-solvus heat treatment is beneficial. Hence, a stepwise heat treatment between solvus and solidus is an effective solutioning approach. This window is generally narrow for superalloys with high refractory content. Hence, stepwise heat treatment within the window would require a large number of incremental temperature steps. Hence, a slow and continuous heating is more practical solutioning method. As experiment 8 is the most effective heat treatment with residual eutectic fraction of less than 1%. Total time required for achieving this solution is approximately 25h over a temperature range of 25°C (1280-1305°C). Hence approximately 1°C/h should be an optimum heating rate of for the continuous heating.

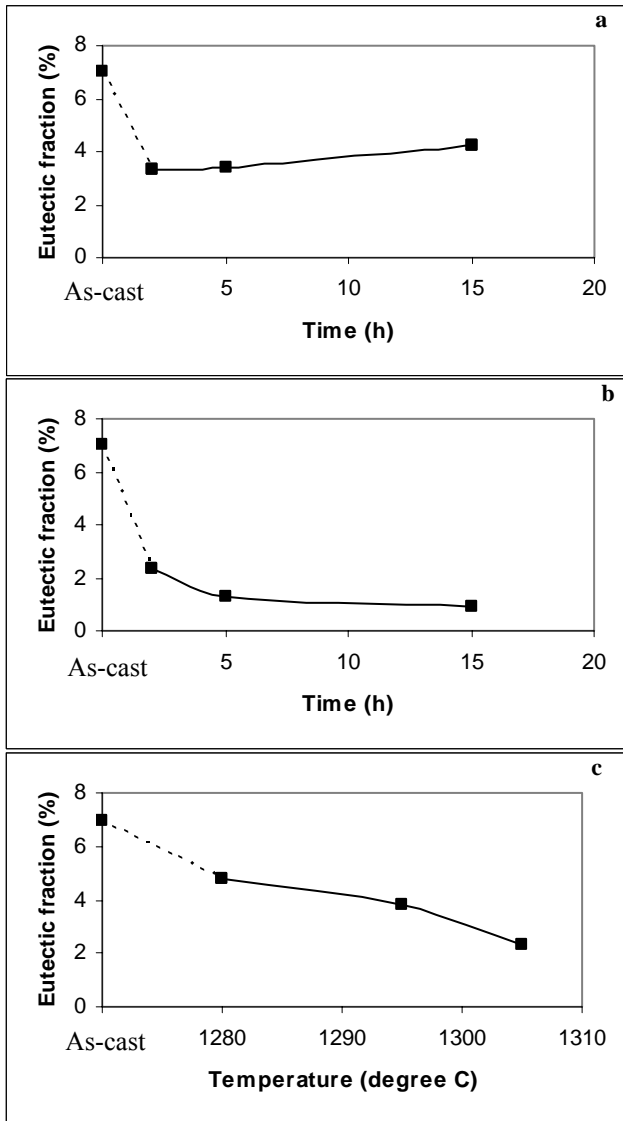


Figure 7: A comparison of variation of eutectic fraction with:
a) holding time at 1280°C from experiments 1,2 and 3.
b) holding time at 1305°C from experiments 6, 7 and 8 and
c) holding temperature from experiment 6.

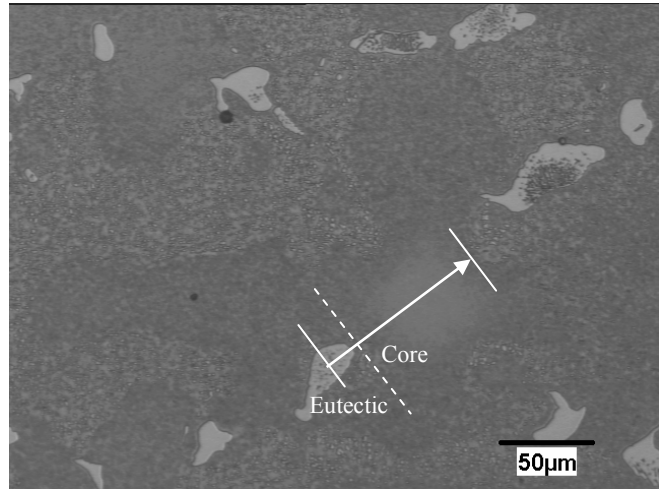


Figure 8: An illustration of EPMA composition profile measurement

Electron Probe Microanalysis (EPMA)

An illustration of EPMA measurements from eutectic region to the dendrite core is shown in Figure 8. The composition profiles of selected elements after heat treatment steps of experiment 6, 7 and 8 are shown in figures 9 and 10. As evident from the figures, there is a sharp gradient between eutectic and core regions. Figure 9 indicates that this gradient decreases with increase in the heat treatment temperature. Conversely, Figure 10 indicates that prolonged holding increases the eutectic to core gradient.

The degree of homogenization can be expressed as the difference between the maximum point and the minimum point on the profile. Lower the difference, flatter the profile, more homogeneous the alloy. Table IV gives variation in degree of homogenization with increase in holding temperature and time. There is a clear trend of increased homogenization with increased heat treatment temperature from as-cast to 1305°C. On the other hand, prolonged isothermal holding from 2h to 20h at 1305°C indicates a decreasing homogenization trend. If the profiles in figure 9 and 10 are carefully observed, it is clear that the dendrite core quickly homogenizes and attains the nominal composition with an increase in temperature. Also, the core remains homogenized during extended isothermal holding. However, during prolonged isothermal holding, the eutectic region diverges from the nominal composition of the alloy. Such an unusual behavior at the eutectic region has been reported as up-hill diffusion [15]. The present work however confirms that up-hill diffusion leads to incipient melting during long isothermal holding (Figure 6d). The results of electron microprobe analysis suggest that stepwise heat treatment with increasing temperature is more beneficial than the isothermal heat treatment.

Table IV: Variation of homogenization with heat treatment

Element	Profile Statistics	Concentration (wt %)				
		As-cast	Expt. 6		Expt. 7	Expt. 8
			1295°C /2h	1305°C /2h	1305°C /10h	1305°C /20h
Al	max	6.13	5.969	5.545	5.98	6.414
	min	4.55	4.668	4.715	4.774	4.711
	Diff	1.58	1.301	0.83	1.206	1.703
Cr	max	8.20	8.067	7.685	7.886	7.695
	min	2.77	3.975	4.651	4.653	2.716
	Diff	5.43	4.092	3.034	3.233	4.979
Ni	max	66.05	65.343	64.492	65.401	67.35
	min	61.87	61.626	61.963	63.155	62.948
	Diff	4.17	3.717	2.529	2.246	4.402
Co	max	5.17	5.261	5.074	5.201	5.216
	min	3.65	4.044	4.27	4.252	3.724
	Diff	1.52	1.217	0.804	0.949	1.492
Ti	max	1.65	1.228	1.229	1.225	1.347
	min	0.66	0.922	0.999	0.977	1.001
	Diff	0.99	0.306	0.23	0.248	0.346
Re	max	2.80	2.6	2.148	2.386	2.261
	min	0.29	0.735	0.946	0.928	0.316
	Diff	2.51	1.865	1.202	1.458	1.945
Ta	max	18.21	15.411	14.457	15.252	17.104
	min	9.41	10.885	11.913	11.89	11.967
	Diff	8.81	4.526	2.544	3.362	5.137
Mo	max	2.08	1.967	1.894	1.881	1.947
	min	0.50	0.878	1.28	0.978	0.399
	Diff	1.58	1.089	0.614	0.903	1.548
W	max	2.83	2.678	2.102	2.263	2.195
	min	0.63	1.313	1.221	1.233	0.82
	Diff	2.20	1.365	0.881	1.03	1.375

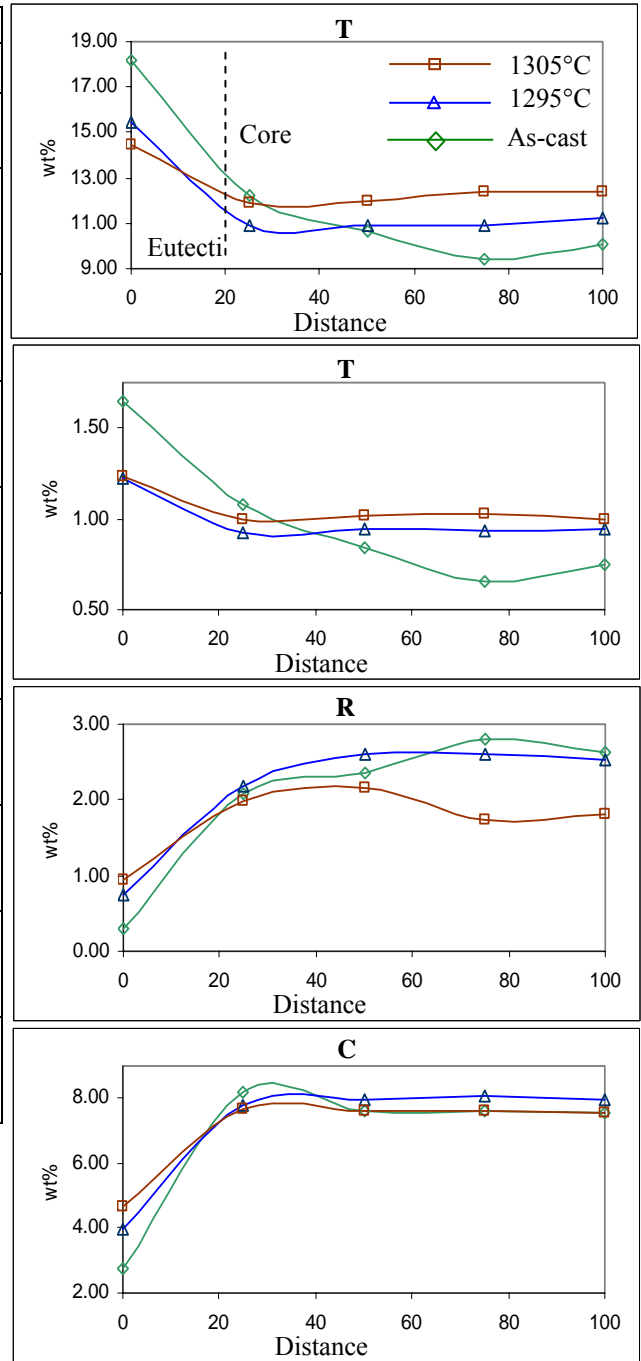


Figure 9: Composition profile from eutectic to core region showing increased homogenization with increased holding temperature

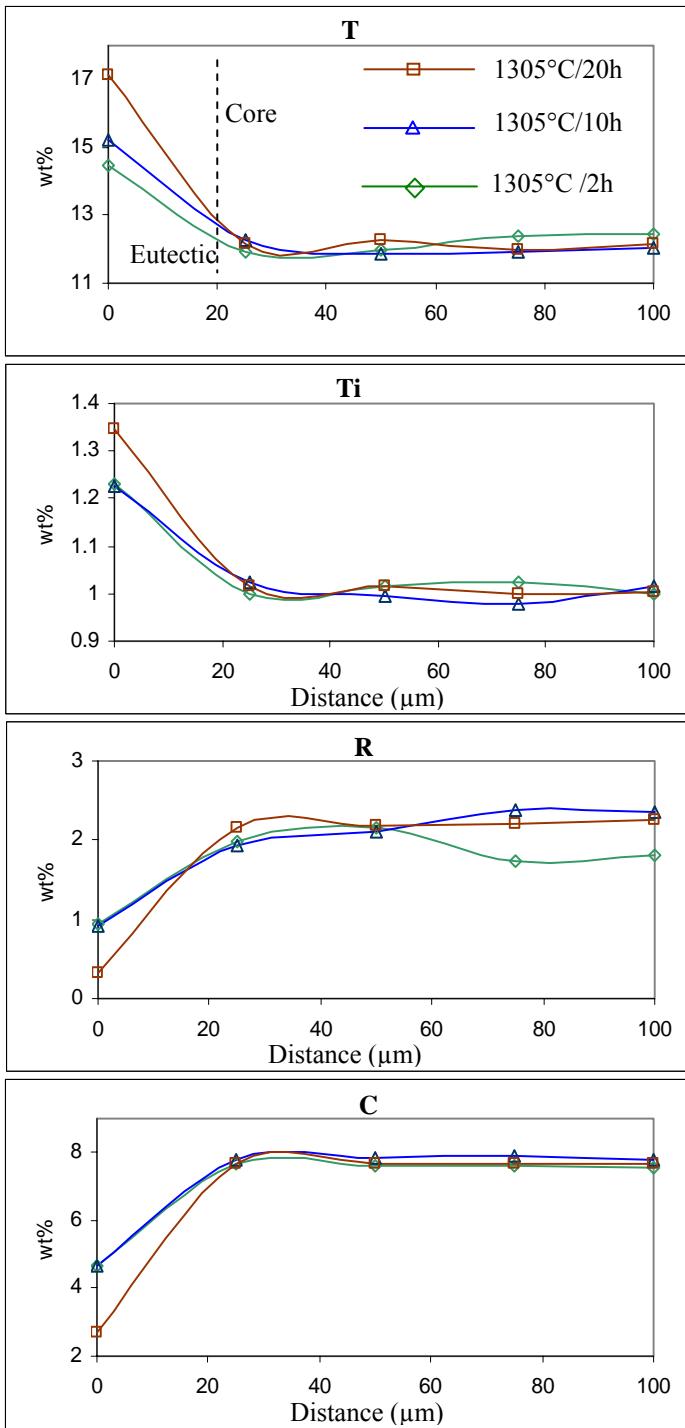


Figure 10: Isothermal composition profiles from eutectic to core region showing a reduction in homogenization with extended holding at 1305°C.

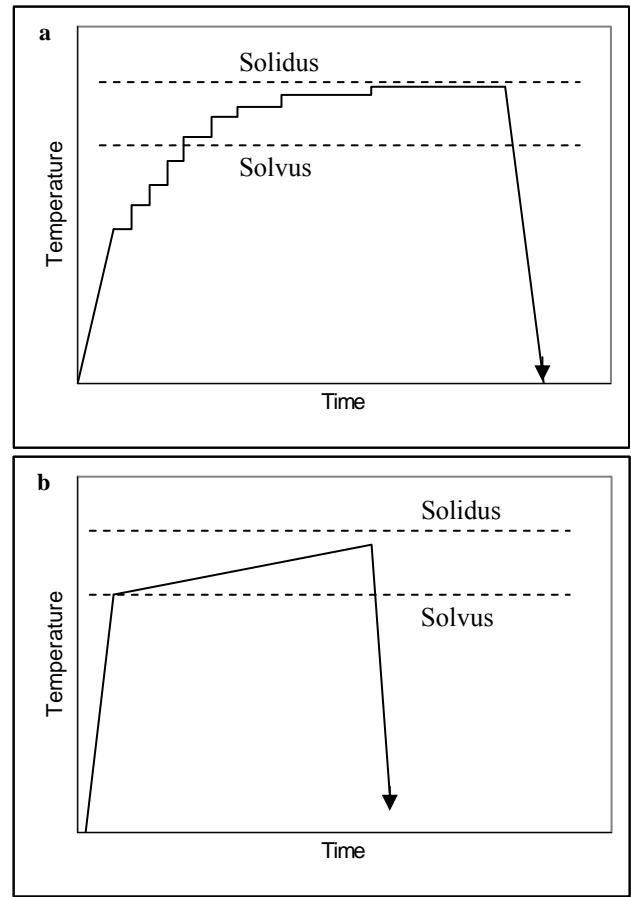


Figure 11: A comparison of a) Traditional step-wise heat treatment and b) proposed heat treatment scheme.

Summary and Conclusions

The heat treatment response of an experimental SX superalloy studied by stepwise heat treatments between 1170 to 1320°C may be summarized as follows.

Sub-solvus homogenization heat treatments help in elevating the incipient melting temperature marginally. Extended isothermal holding at a sub-solvus temperature has no beneficial effect on the dissolution of eutectic γ' . Extended holding at and above solidus temperature leads to incipient melting, irrespective of the thermal history of the alloy.

Both extended holding and isothermal holding between solvus and solidus temperatures homogenizes the dendrite core quickly. However, eutectic region indicate up-hill diffusion during isothermal holding leading to incipient melting. Hence, continuous heating at a slow heating rate between the solvus and solidus seems to be the favorable condition for the complete solutioning and homogenization of the alloy without incipient melting. Hence a new solutionizing-homogenizing heat treatment scheme, compared to the usual traditional stepwise heat treatment, shown in Figure 11 is proposed.

The procedure for designing an effective homogenizing-solutioning heat treatment for a newly designed alloy with heavy refractory content would be:

1. Decide the temperature range of interest from the DSC thermograph of the as-cast alloy.
2. Determine the approximate solvus and solidus temperatures by stepwise heat treatments with increasing temperature and the constant holding time of 2h.
3. Quickly raise the temperature of the specimen to the solvus temperature.
4. Heat the specimen at a slow rate of about 1°C/h.
5. Quench just before reaching the solidus temperature.

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