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Research Article

Effect of Homogenization on Microstructural Refinement and Hot Ductility René65 Nickel-Based Super Alloys

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ABSTRACT

In this study, the effect of different homogenization treatments on the Rene65 nickel-based super alloy was investigated. Single-step homogenization was performed at two temperatures, 1100°C and 1150°C, each for a duration of 5 hours. Additionally, a two-step homogenization cycle consisting of 5 hours at each temperature was conducted. The Hot ductility behavior was evaluated through hot tensile test at various temperatures. The results demonstrated that single-step homogenization at the higher temperature of 1150°C led to lower ductility (%10) and brittle fracture characteristics, in comparison to the 1100°C treatment (%45). This reduced ductility is likely due to the presence of low-melting-point secondary phases enriched in zirconium, which promote grain boundary weakening and intergranular failure during high-temperature deformation. In contrast, the two-step homogenization (initially at 1100°C followed by 1150°C), followed by air cooling, resulted in significantly improved ductility (%45) compared to single-step high-temperature homogenization. This enhancement in ductility may be attributed to the partial retention of interdendritic phases—such as eutectic structures and blade-like precipitates (possibly η-phase)—which did not fully dissolve during homogenization or re-precipitated during rapid cooling. These retained features may act to inhibit excessive grain growth and suppress grain boundary embrittlement.

1. Introduction

Nickel-based superalloys are essential for high-temperature applications, particularly in turbine disks, where they are exposed to severe mechanical and thermal loads. Among these alloys, René 65—a cast-wrought alloy derivative of René 88DT—has become widely used as a cost-effective alternative to powder-metallurgy superalloys.

Rene65 Nickel-based super alloy providing comparable mechanical performance at temperatures above 700°C. The alloy's microstructure consists of a γ (gamma) matrix strengthened by γ′ (gamma-prime) precipitates, which play a key role in maintaining high-temperature strength. However, the cast-wrought processing route leads to microsegregation of alloying elements (e.g., Ti, Nb, W, and Mo) during solidification.

This segregation promotes heterogeneous microstructures that reduce hot ductility and workability.

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The resulting localized strain hardening increases the flow stress and limits deformability. Therefore, homogenization heat treatments are essential for dissolving detrimental phases (such as γ–γ′ eutectics, carbides, low-melting-point and other TCP phases) and improving hot workability prior to subsequent thermo-mechanical processing [1, 2].

Multistage homogenization, with gradual temperature ramping, further reduces the risk of incipient melting in alloys with high segregation levels. Segregation of elements such as Zr can form enriched phases that initiate melting within this temperature range, similar to what has been reported in alloys like Udimet720Li and IN939. Several studies have also documented low-melting constituents—particularly Zr-rich and B-rich phases—in nickel-based superalloys such as IN718, Waspaloy, and René 41. These phases can significantly affect the homogenization response at elevated temperatures, especially under single-step heat treatments, and may promote localized melting that reduces hot workability and degrades subsequent thermo-mechanical processing [2–6].

Research on the Rene65 superalloy remains limited. To date, only a single study has addressed its homogenization treatment, and the investigation provides little detailed insight into the influence of segregation on high-temperature ductility.

According to the findings of Lang Shui and Jianhui Fu, a heat-treatment temperature of 1160°C for 50 hours was identified as an effective homogenization condition for René 65 billets with a diameter of 250mm [4].

However, their study did not address the potential for incipient melting or the presence of low-melting-point phases. Therefore, in this study, homogenization was performed using both single-step and two-step high-temperature treatments in the range of 1100 to 1150°C. The primary objective was to dissolve and deep investigate undesirable phases—including γ–γ′ eutectics, eta phase, and Zr-rich phases—and to evaluate their influence on hot ductility.

2. Material and Method

The material used in this study was as-cast René 65 super alloy, produced via a double-melting process (VIM[1]/ESR[2]) to ensure high purity and homogeneity. The nominal chemical composition of the alloy is shown in Table 1.

To investigate the effect of homogenization on microstructure and hot ductility, different single and two step homogenization treatments were performed (Table 2.).

Samples were sectioned using a precision cutting machine to avoid microstructural damage. Then ground with SiC abrasive papers (up to 2500 grit), and polished using 1 μm diamond suspension. Marble's reagent (10g $CuSO_4$ + 50ml HCl + 50ml H_2O) was used for general microstructure observation.

Modified reagent (5g $FeCl_3$ + 25ml HCl + 25ml H_2O) was applied to highlight inter-dendritic phases and precipitates. Optical microscopy (OM, Zeiss Axio Observer) and scanning electron microscopy (SEM, with EDS) were employed to analyze Fracture Surfaces and Dendritic segregation and precipitate distribution in inter-dendritic regions. Tensile specimens were machined parallel to the solidification direction then were tested at temperatures of 1050°C, 1075°C, 1100°C, and 1150°C in accordance with ASTM E21 with Istron8502 machine at a constant strain rate of 0.01 s^{-1}.The specimens were initially heated at a controlled rate of 5°C/s to the target temperature, followed by a 10-minute hold to ensure thermal equilibrium.

Table 1. Chemical composition of Rene65 (wt%).

Cr	Co	Mo	W	Ti	Al	Fe	Nb	Zr	B	C	Ni
16	13	4	4	3.7	2.1	0.8	0.7	0.05	0.015	0.0	Bal

Table 2. Homogenization Processes.

Process	Temperature(°c)	Time(h)	Cooling	Temperature(°c)	Time(h)	Cooling
One step	1100	5	Air	-	-	-
One Step	1150	5	Air	-	-	-
One Step	1100	5	Air jet	-	-	-
Two Step	1100	5	Air jet	1150	5	Air
Two Step	1100	5	Air	1150	5	Air
*Heating rate: 10°C/min to ensure uniform temperature distribution. *Temperature control accuracy: ±3°C.						

1- Vacuum Induction Melting
2- Electro-Slag Remelting

3. Results and Discussion

Fig. 1. presents the engineering stress–strain curves of specimens homogenized in a single-step process at 1100°C and 1150°C. As shown in the figure, the sample homogenized at 1150°C exhibits lower elongation to failure, indicating reduced ductility. In contrast, the specimen homogenized at 1100°C demonstrates significantly higher ductility, with an elongation of approximately 48% prior to fracture. Fig. 2. shows the corresponding fracture surface images. The specimen homogenized at 1150°C displays features characteristic of brittle fracture, such as inter-granular cracking and cleavage-like morphology. The solidification structure of the as-cast alloy reveals that the inter-dendritic regions, being the last to solidify, are highly enriched in solute elements due to segregation. These regions are potential sites for the formation of various secondary phases, including eutectic constituents, carbides, solute-rich intermetallic, and potentially brittle topologically close-packed (TCP) phases. The presence and evolution of these phases during homogenization and high-temperature deformation may contribute to reduced ductility and increased susceptibility to inter-granular fracture in samples treated at higher homogenization temperatures.

Scanning Electron Microscopy (SEM) analyses (Fig. 3.) indicated the presence of zirconium-rich precipitates at grain boundaries. These precipitates are believed to promote local melting during high-temperature single-step homogenization treatments, thereby increasing the susceptibility to grain boundary liquation and intergranular fracture. Fig. 2. shows an SEM micrograph of zirconium-rich precipitates. EDX analysis confirms that these precipitates contain approximately 13% wt zirconium, suggesting that they are most likely of the (Ni, Cr)5 Zr type. These intermetallic phases are presumed to have formed during solidification, predominantly within interdendritic regions and along grain boundaries.

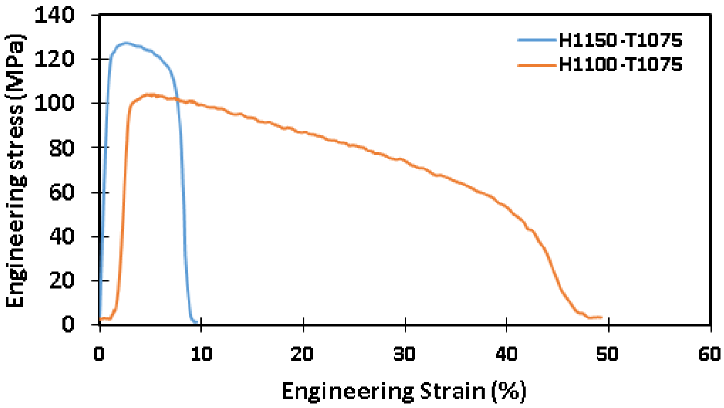


Fig. 1. The flow curves of homogenized specimens treated at 1100°C and 1150°C, obtained from hot tensile tests conducted at 1075°C and a strain rate of 0.01 s^{-1}.

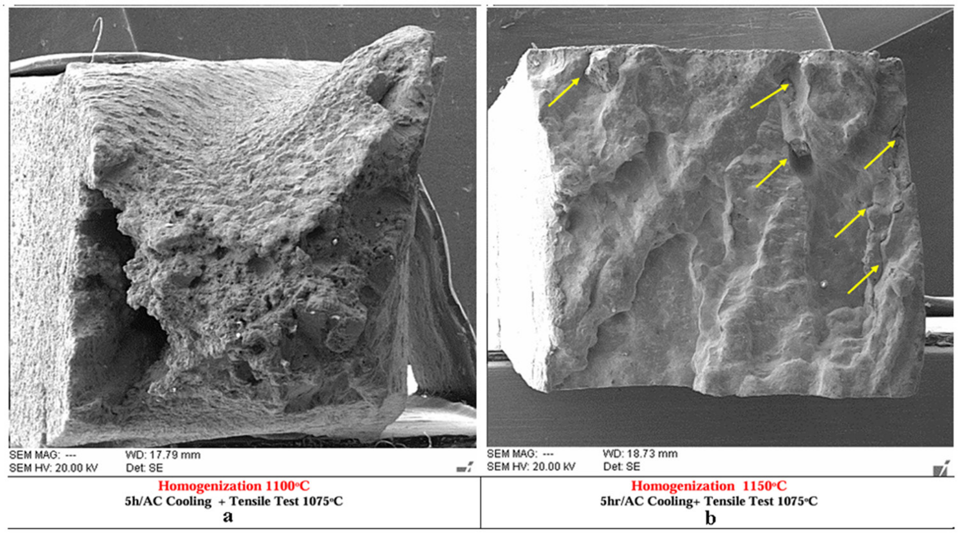
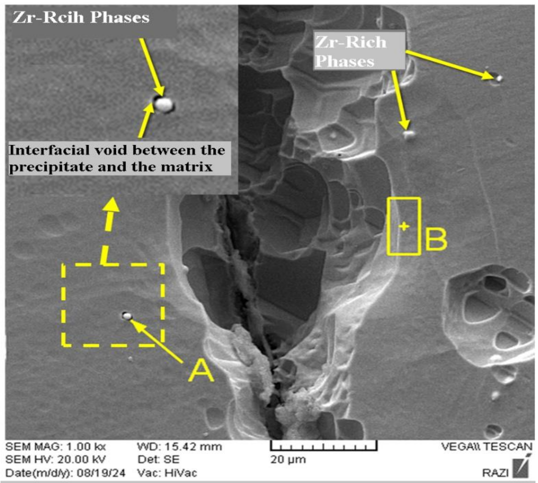


Fig. 2. Fracture surface morphologies of specimens subjected to hot tensile testing at 1100°C and a strain rate of 0.01s^{-1}: (a) Homogenized at 1100°C showing predominantly ductile features with localized inter-granular cracking; (b) Homogenized at 1150°C exhibiting more pronounced inter-granular fracture.

Guang-Di Zhao,Lian-Xu Yu also showed that the final liquid is mainly constituted by Ni and Zr, and Ni_5Zr is formed at the end of solidification Udimet720Li and The melting point are between 1130 and 1140ºC for Ni_5Zr phase. Jahangiri, Boutorabi Studied on incipient melting in cast Ni base IN939 super alloy during solution annealing and its effect on hot workability. Standard temperatures of solution annealing (i.e. 1150–1160ºC) caused local melting that occurs in the regions around the Eta phase. The incipient melted regions enriched in Zr, Nb and B elements contained three different phases after water quenching. Small amounts of incipient melted regions impair hot workability of the IN939 super alloy. Results showed long time annealing at 1150ºC or using a two-step heat treatment can lead to significant improvement in hot workability of the alloy [7-13].



Spectra: ZONE B.spx

Element	Series	unn. C [wt.-%]	norm. C [wt.-%]	Atom. C [at. -%]
Aluminum	K series	1.97	2.42	5.22
Titanium	K series	2.07	2.54	3.09
Chromium	K series	10.67	13.08	14.67
Iron	K series	0.51	0.63	0.66
Cobalt	K series	10.42	12.78	12.64
Nickel	K series	46.22	56.66	56.29
Zirconium	L series	3.25	3.98	2.54
Niobium	L series	3.25	3.99	2.50
Molybdenum	L series	3.20	3.93	2.39

Spectra: ZONE A.spx

Element	Series	unn. C [wt.-%]	norm. C [wt.-%]	Atom. C [at. -%]
Aluminum	K series	1.24	2.04	4.54
Titanium	K series	2.58	4.23	5.31
Chromium	K series	8.53	14.03	16.20
Cobalt	K series	5.52	9.07	9.24
Nickel	K series	30.36	49.93	51.08
Zirconium	L series	12.59	20.70	13.63

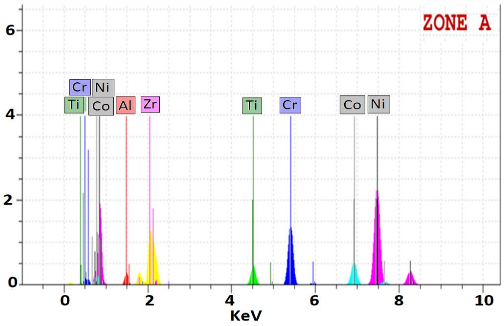


Fig. 3. SEM image of a grain boundary region in the specimen homogenized at 1150°C. Zr-riche phases (white spots) near grain boundary and interior of fracture dimples.

Fig. 4. presents the high-temperature stress-strain curves at various test temperatures for the sample homogenized at 1100°C and air-jet cooled. As evident from the Fig. 4. the specimen tested at 1100°C demonstrates superior elongation characteristics. Fig. 5. and Table 3. illustrates the stress-strain curves and reduction area for three different homogenization treatments respectively. Hot tensile tests were conducted at 1100°C with a strain rate of $0.01s^{-1}$. As can be observed from Fig. 1. the sample subjected to single-stage homogenization at 1100°C for 5 hours, followed by air-jet cooling, exhibits higher strength and lower elongation compared to the other variants. The sample homogenized at 1100°C for 5 hours and cooled via air-jet, followed by homogenization at 1150°C for 5 hours with air cooling, exhibits improved elongation. Additionally, the sample homogenized at 1100°C, air jet-cooled, and then re-homogenized at 1125°C for 5 hours with air cooling shows a lower enhancement in elongation, accompanied by a marginal decreasing in stress level.

Fig. 6. shows optical micrographs and SEM of the alloy structure homogenized at 1100°C for 5 hours and subsequently air jet cooling. As observed, the grain boundaries and interdendritic regions contain needle-like or plate-like precipitates, which are distributed along the dendritic network. Additionally, unresolved eutectic structures are still present between the needle-shaped precipitates in these regions, indicating incomplete dissolution of interdendritic phases during the homogenization process. The relatively lower elongation observed in samples homogenized at 1100°C through a single-step process, compared to those subjected to two-step homogenization, may be attributed to the presence of residual, undissolved phases in the interdendritic regions. These retained phases, such as eutectic constituents or plate-like intermetallic, can act as stress concentrators and limit ductility during high-temperature deformation.

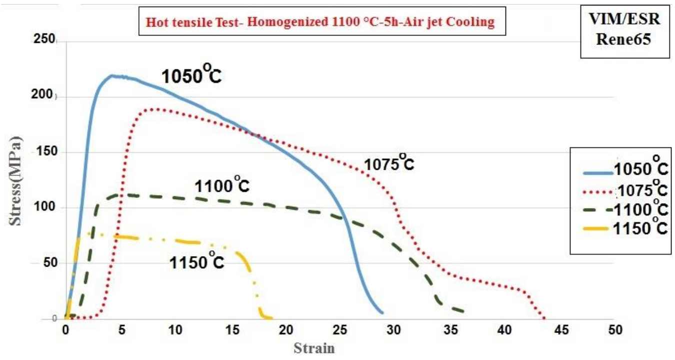


Fig. 4. Stress–strain curves at various temperatures during hot tensile testing at a strain rate of $0.01s^{-1}$, after homogenization at 1100°C followed by air-jet cooling.

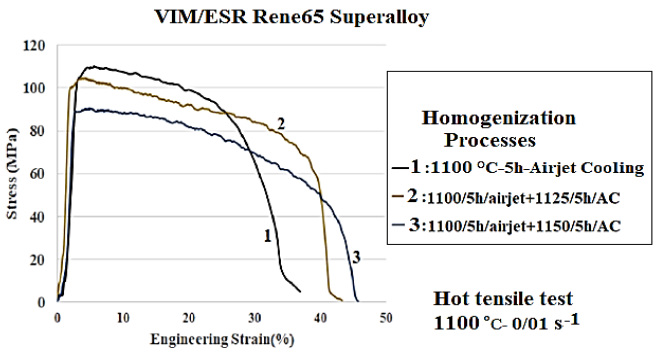


Fig. 5. The stress-strain curves obtained at 1100°C with a strain rate of $0.01s^{-1}$ for samples subjected to single-stage and two-stage homogenization treatments.

Table 3. Reduction Area for different Homogenization Process of Rene65 test at 1100°C and strain rate of $0.01s^{-1}$.

Homogenization process	Reduction Area(%)
1100/5h/ air jet cooling	56
1100/5h/airjet+1125/5h/AC	69
1100/5h/airjet+1150/5h/AC	71

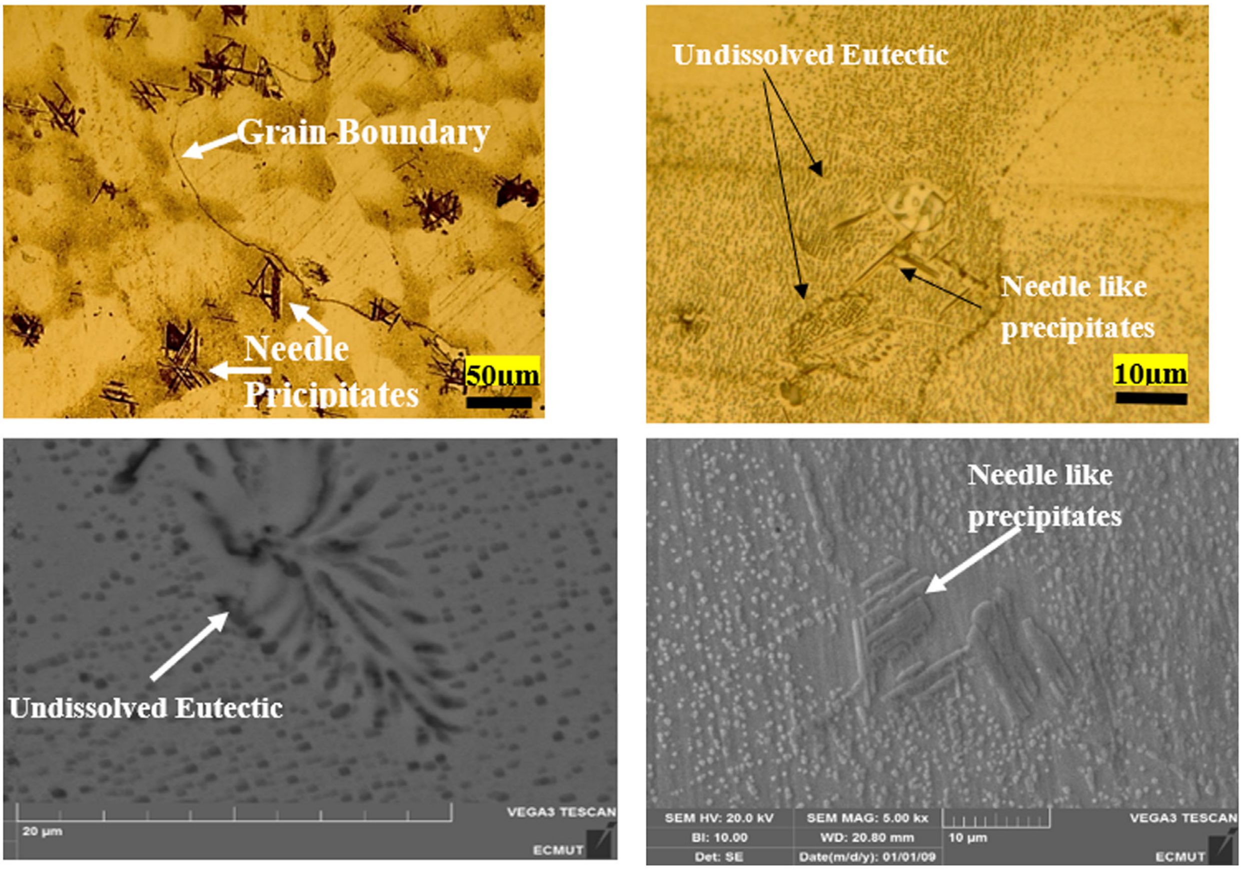


Fig. 6. Optical and SEM micrographs of the interdendritic regions shows undissolved phases in the specimen homogenized at 1100°C and air jet cooled.

The line scan (Fig. 7.) reveals elemental segregation patterns, indicating enrichment of titanium and possibly other alloying elements such as aluminum or niobium, which suggests that the precipitates may correspond to the η-phase or other intermetallic compounds formed during or after homogenization. The η-phase can nucleate near grain boundaries, within grains, or adjacent to MC-type carbides. In addition, the transformation of γ' to η can also occur more readily in low-carbon super alloys. At a temperature of approximately 1100°C—close to the dissolution temperature of primary γ' precipitates—titanium may be released from dissolving γ' or eutectic phases. If the carbon content is insufficient to form or coarsen MC carbides, this liberated titanium may instead promote the precipitation of the η-phase. This phase transformation pathway becomes more favorable under such conditions, especially in the presence of solute segregation in interdendritic or grain boundary regions. the formation energy of L1 2 Ni 3 Ti. The OA-γ' structure is higher than D0 24 for is precipitated as an intermediate phase for its low nucleation barrier but transforms to η thermodynamically stable [13-16].

The two-step homogenization process, involving initial soaking at 1100°C followed by a secondary stage at 1150°C, significantly influenced the microstructural evolution of the nickel-based super alloy. As observed in Fig. 8. Sample rolled without cracks in edges with two step homogenization. the interdendritic regions in the two-step homogenized sample are notably free of blade-like (needle-shaped) precipitates, which were otherwise present in the single-step homogenized specimens at 1100°C. The absence of these precipitates can be attributed to the more gradual dissolution of low-melting interdendritic phases during the first stage at 1100°C, preventing the onset of localized melting that may otherwise occur during direct high-temperature homogenization. Moreover, the slower kinetics of η-phase or other intermetallic formation at this initial stage help reduce solute segregation and promote more uniform phase distribution. Consequently, this homogenization route likely suppresses the formation of detrimental phases such as η or TCP (topologically close-packed) structures in the interdendritic regions. This microstructural refinement enhances the hot ductility of the alloy, as also confirmed by the higher elongation observed in tensile testing compared to the single-step homogenized counterparts. These findings suggest that optimized multi-step homogenization schedules can be a key strategy to improve the process ability and performance of advanced nickel-based super alloys.

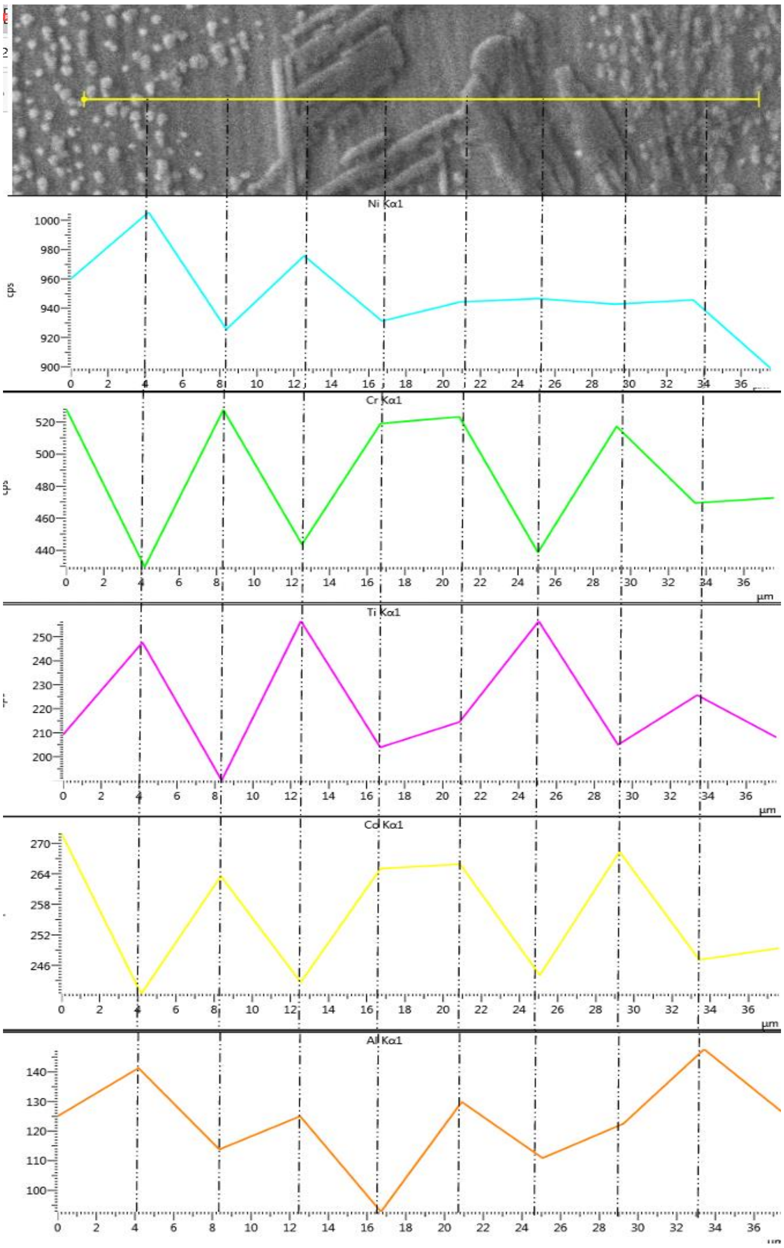


Fig. 7. Linear EDS analysis across the blade-like precipitates.

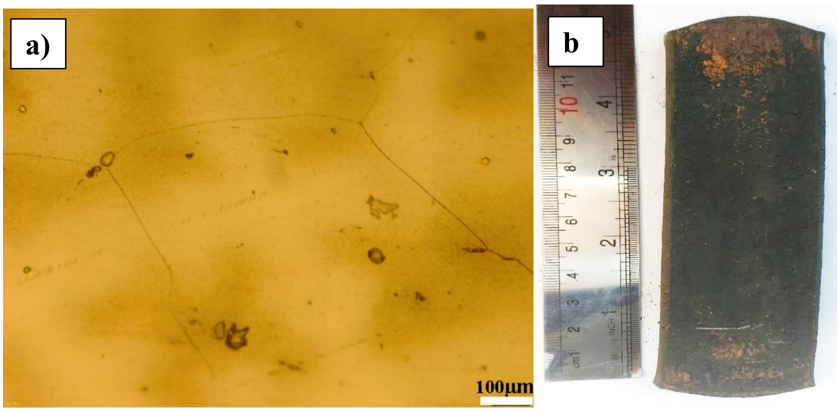


Fig. 8. a)Microstructural morphology of the interdendritic regions of the two-step homogenized sample at 1100°C /5h+ 1150°C /5h, b) As rolled Rene65 after two step homogenization at 1100°C.

4. Conclusions

- The single-step homogenization at 1100°C exhibited a higher elongation (%10) compared to homogenization at 1150°C.
- At the homogenization temperature of 1150°C, phases enriched with zirconium (Ni_5Zr) were detected along the grain boundaries, which may act as localized melting points within the homogenization temperature range (1130-1160).
- At temperatures close to the dissolution temperature of the gamma prime (γ′) precipitates, due to the low carbon content in the alloy, the formation of eta (η) phases in the microstructure in lower time homogenization (5h) is possible as a result of localized increases in titanium concentration.
- The two-step homogenization process involved an initial treatment at 1100°C for 5 hours followed by air cooling, and then a subsequent homogenization at 1150°C have better ductility and workability in high temperature than 1100°C

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