

**COMPARATIVE STUDY OF HEAT TREATMENT CYCLES IN  
PRECIPITATION HARDEN NIMONIC 80A SUPERALLOY**Jaimin Panchal<sup>1</sup>, Prof. M.N. Patel<sup>2</sup><sup>1</sup>*Metallurgy and Materials Engg. Dept., The Maharaja Sayajirao University Of Baroda,  
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**Abstract** - Super alloy Nimonic 80A is the alloy from series NIMONIC nickel-base superalloys that contain 20% of chromium it is intended for use at elevated and high temperatures at which the creep process occurs significantly. The primary strengthening mechanism of this super alloy is based on precipitation of fine and coherent particle of intermetallic  $\gamma'$  phase  $Ni_3(Al,Ti)$ , which ensure needed creep strength. This strengthening mechanism for such super alloy is more favorable in relation to other strengthening mechanisms. The effect of hardening that can be achieved by  $\gamma'$  phase depends on amount, dispersion, and size of  $\gamma'$  phase. All mentioned is controlled by heat treatment. In the present work an attempt has been made to study the effect of two different heat treatment cycles on the microstructures of super alloy Nimonic-80A.

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**Keywords** – Nimonic 80A, Heat treatment, continuous carbide network, gamma prime precipitates, long term creep rupture properties.

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**I. INTRODUCTION**

Nimonic 80A is a trade name of Special Metals Corporation and also designated as UNS N07080 – which is a nickel based super alloy. The high temperature stability of nickel based material can greatly be enhanced by forcing precipitation of finely dispersed second phase in the solid solution of nickel, the term referred as solid solution hardening. The solid solution hardening is achieved through solution annealing heat treatment for UNS N07080 which creates imperfection in atomic arrangement caused by dissolution of one element in another. This is known as dislocation that imparts atomic level plastic deformation to metal and in turn it requires higher force for dislocation movement at atomic level. Any disturbance in crystal lattice that hinders dislocation movements thus reduces rate of plastic deformation and finally increases the strength of material. All such atomic level imperfections are stronger than pure metal. The additive effect increases the strength of material up to an optimum level until the single phase solid solution exists. The maximum strengthening is obtained when large number of, finely dispersed elements are dissolved to form an extremely complex solid solution in base metal. The presence of 18-21% chromium makes the material superior through solid solution strengthening suitable for low creep strain and corrosion resistant.[1,2,10] Additionally, UNS N07080 obtains superior strength at elevated temperature through precipitation hardening mechanism. Presence of titanium and aluminium acts as precipitation hardening elements. The strengthening due to precipitation hardening is achieved through fine dispersion of second phase within the solid solution of nickel-chromium matrix and is generally achieved with two heat treatment steps.[1,2,11]

**II. EXPERIMENTAL PART**

The project work started with as received Nimonic 80A samples from an industry. Two heat treatment cycles were carried out on separate Nimonic 80A samples to find out the detailed microstructural analysis, which further helps in improving creep properties [1,3-6]. The experimental work was done in the following steps.

- Chemical analysis
- Microstructure evaluation
- Heat treatment
- Scanning electron microscopy
- Energy dispersed spectroscopy

**2.1 CHEMICAL ANALYSIS**

The chemical analysis was carried out by optical emission spectrometer. Here each and every element present in the Nimonic alloy was qualified accurately. The following table shows the results.

**Table 1 : Result obtained through Optical Emission Spectroscopy**

<b>ELEMENTS</b>	<b>MEASURED</b>	<b>REQUIRED (ASTM B 637 UNS NO7080)</b>
Carbon (%)	0.021	0.10max.
Sulphur (%)	0.002	0.015max.
Phosphorous (%)	0.010	-
Manganese (%)	0.00	1.00max.
Silicon (%)	0.0212	1.00max.
Chromium (%)	19.82	18.00-21.00
Nickel (%)	74.80	-
Molybdenum (%)	0.142	-
Aluminium (%)	1.30	0.50-1.80
Iron (%)	0.940	3.00max.
Niobium (%)	0.047	-
Titanium (%)	2.39	1.80-2.70

## 2.2 HEAT TREATMENT

### Heat-Treatment cycle I

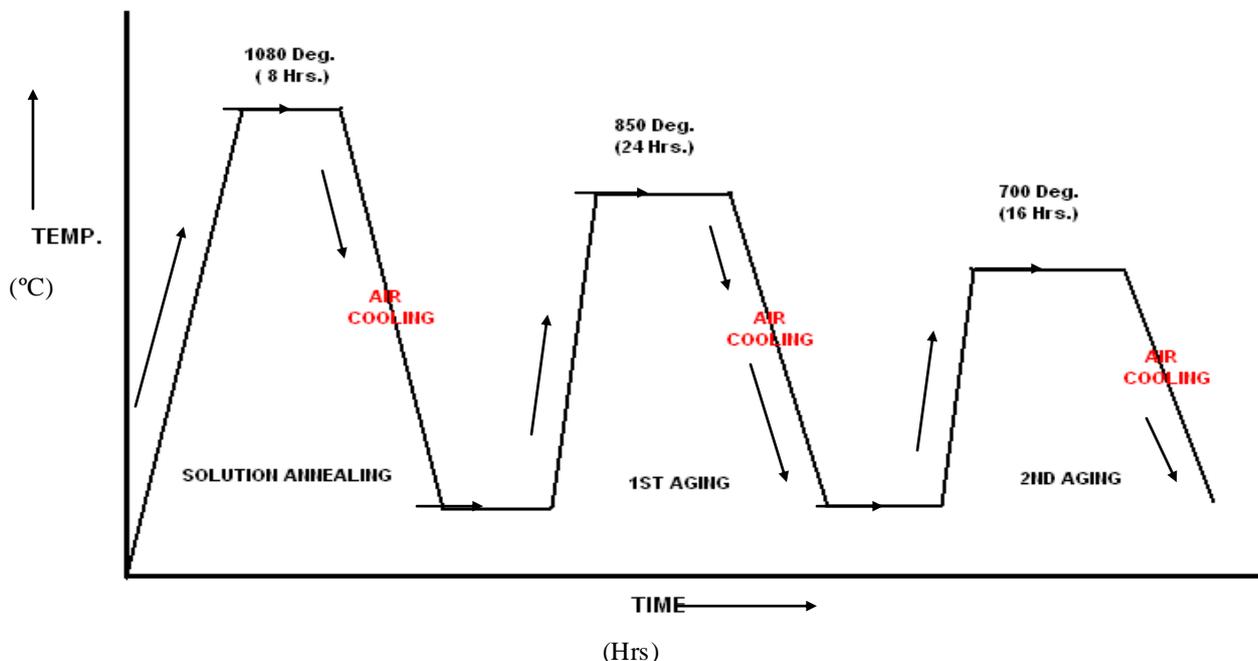
**SOLUTION ANNEALING:** The solution annealing was carried out at 1080°C for 8hours and then air cool.

**AGING:**

First aging was carried out at 850°C for 24 hours and then air cool.

Second aging was carried out at 700°C for 16 hours and then air cool.

**Heat Treatment Graph:** X-axis – Time (hrs) ; Y-axis – Temperature(°C).



**Figure 1. Heat Treatment Graph 1 for Heat Treatment cycle I**

### Heat-Treatment cycle II

**SOLUTION ANNEALING :** The solution annealing was carried out at 1100°C for 8 hours and then transfer direct to the furnace at 850°C and hold for 24 hours, and then furnace cool .

Then, aged at 700°C for 16 hours and then air cool.

Heat Treatment Graph: X-axis – Time(hrs) ; Y-axis – Temperature(<sup>0</sup>C).

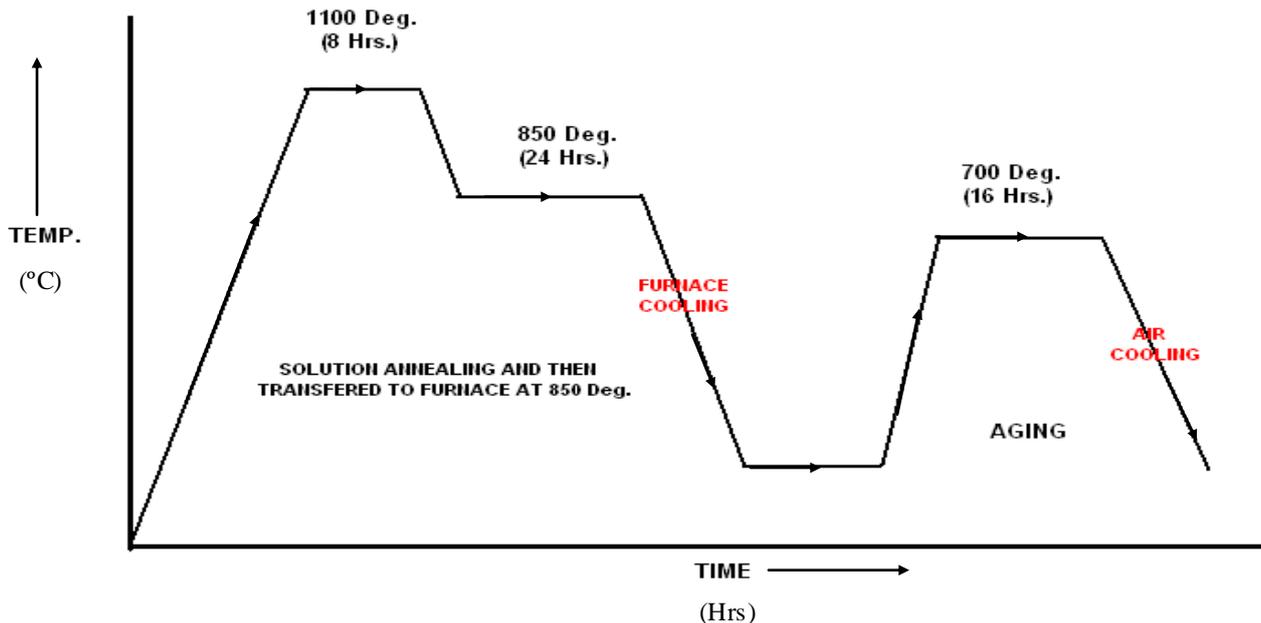


Figure 2. Heat Treatment Graph 2 for Heat Treatment cycle II

### 2.3 MICROSTRUCTURE EVALUATION

The microstructures of two different heat treatment cycle samples were taken for examination. The microstructure was developed as per ASTM 923-03. The test method includes

#### PREPARATION OF TEST SPECIMEN:-

From samples, separate coupons were cut for longitudinal and transverse sections. Because high temperature or mechanical deformation associated with particular cutting processes can alter the structure of the alloy. The cutting of the specimen should be by a technique that prevents these effects. Cross sectional surface were polished to a metallographic finish suitable for examination on optical microscope after etching. The polishing was carried out on successively finer emery papers 1/0, 2/0, 3/0 and 4/0 and then to the polishing clothe with alumina ( $Al_2O_3$ ).

#### ETCHING SOLUTION:-

The solutions for etching were Oxalic acid,  $H_2O_2+HCl$ , Kallings reagent.

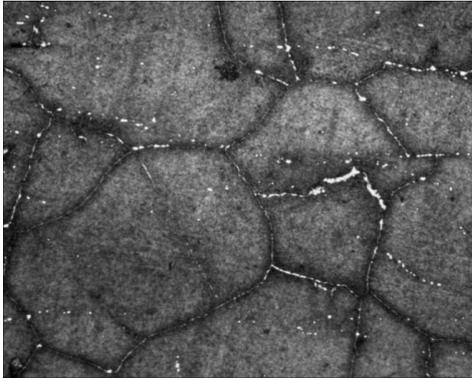
#### ETCHING CONDITION:-

The polished specimens were etched at 3 V dc, for 60 seconds. After etching, the specimen was rinsed with water and dried in air.

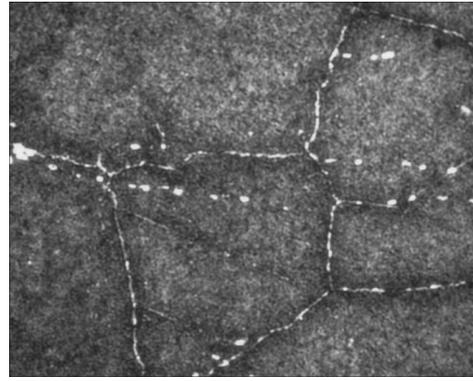
*Metallographic prepared sections were initially examined in an optical microscope and, subsequently, evaluated in a scanning electron microscope equipped with an EDS spectrometer.*

## III. NIMONIC-80A: TEST RESULT AFTER HEAT TREATMENT CYCLE I

### 3.1 MICROSTRUCTURE EXAMINATION (OPTICAL MICROSCOPE)



**Figure 3. (400X)**

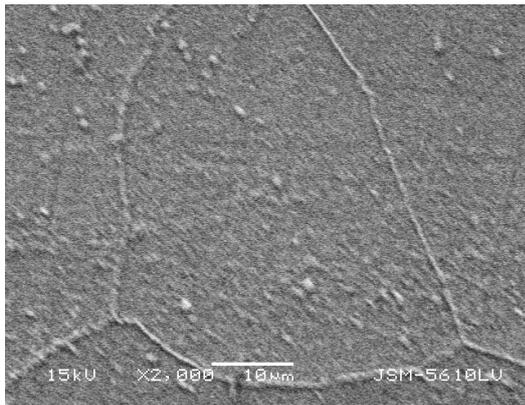


**Figure 4. (1000X)**

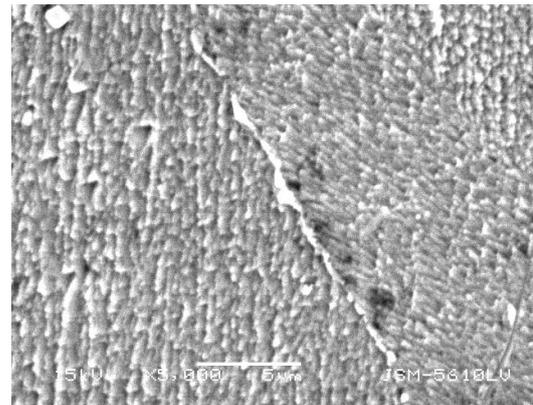
Figure 3. & Figure 4. Shows: Microstructure examination revealed presence of continuous carbide network, which can affect the long term creep properties.

### 3.2 MICROSTRUCTURE EXAMINATION (SCANNING ELECTRON MICROSCOPE)

#### SEM ANALYSIS OF HEAT TREATMENT CYCLE I SAMPLE



**Figure 5. (5000X)**



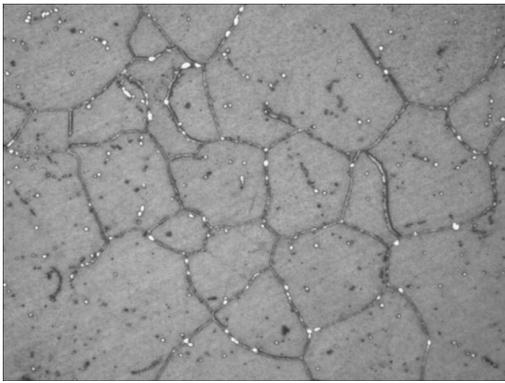
**Figure 6. (5000X)**

Figure 5. shows: show carbides precipitation in grain boundaries that is represented in the formation of continuous films (including 20.05 percent Cr) and dispersed particles (include 3.15 percent Ti) of carbides, respectively. Carbides precipitation results in decreasing of alloy ductility and toughness.

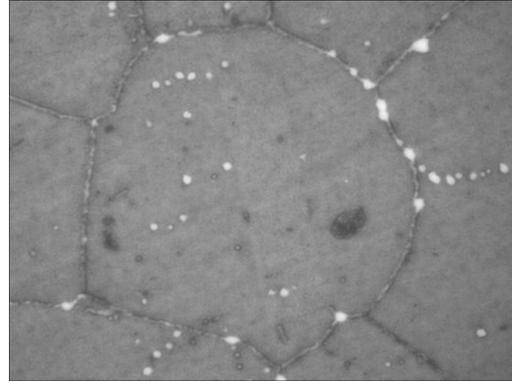
Figure 6. Shows: distribution and morphology of strengthening phase  $\gamma'$  (Gamma Prime) precipitates.

## IV. NIMONIC-80A: TEST RESULT AFTER HEAT TREATMENT CYCLE II

### 4.1 MICROSTRUCTURE EXAMINATION (OPTICAL MICROSCOPE)



**Figure 7. (400X)**

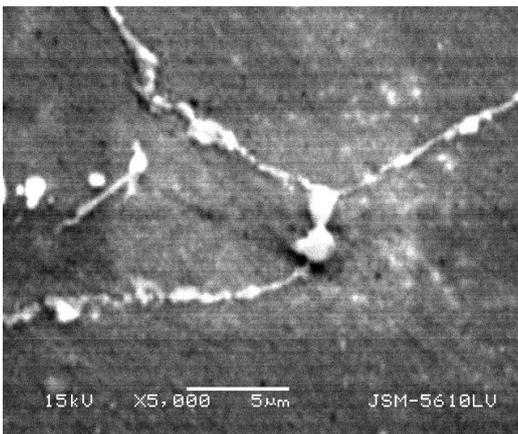


**Figure 8. (1000X)**

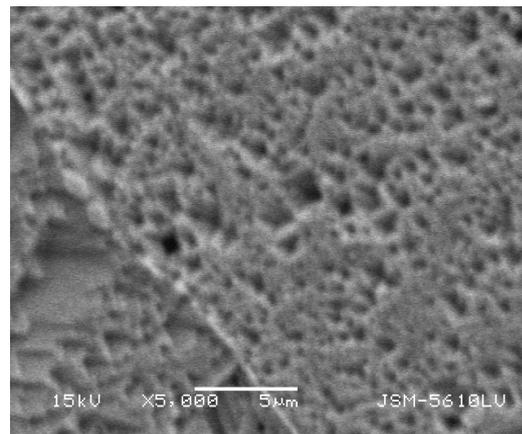
Figure 7. & Figure 8. Shows: Microstructure examination revealed that continuous carbide network is getting dissolved and started disappearing.

#### 4.2 MICROSTRUCTURE EXAMINATION (SCANNING ELECTRON MICROSCOPE)

##### SEM ANALYSIS OF HEAT TREATMENT CYCLE II SAMPLE



**Figure 9. (5000X)**



**Figure 10. (5000X)**

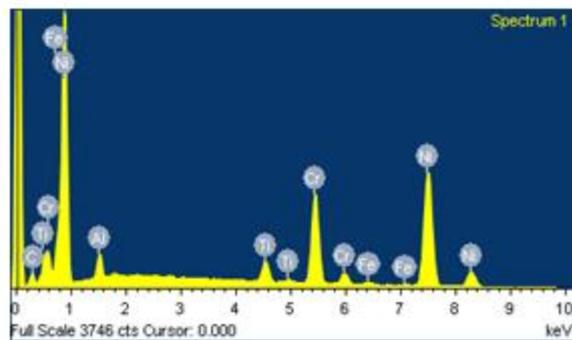
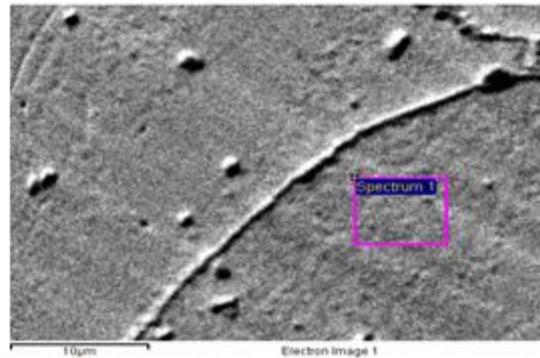
Figure 9. shows: SEM examination revealed that continuous carbide network is getting dissolved and started disappearing.

Figure 10. shows: further refinement in distribution and morphology of strengthening phase  $\gamma'$  (Gamma Prime) precipitates.

#### 4.3 ENERGY DISPERSIVE SPECTROSCOPY MICROANALYSIS

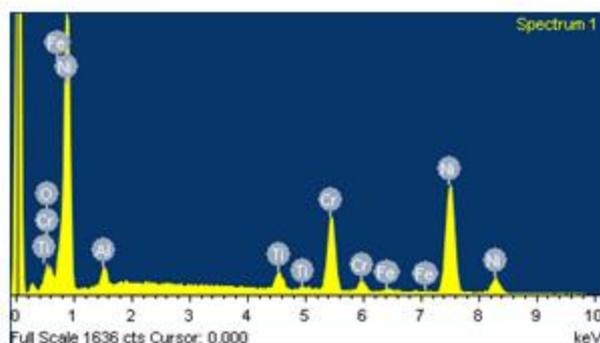
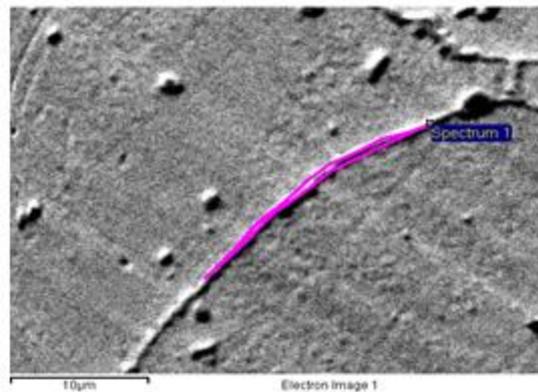
### EDS REPORT – Austenitic Matrix

Element	Weight%	Atomic%
CK	5.01	19.55
AlK	2.14	3.72
TiK	3.15	3.08
CrK	20.05	18.05
FeK	1.17	0.98
NiK	68.47	54.61
Total	100.00	



### EDS REPORT – Grain Boundary

Element	Weight%	Atomic%
OK	1.15	3.89
AlK	1.94	3.89
TiK	3.05	3.44
CrK	18.58	19.33
FeK	0.99	0.96
NiK	74.30	68.48
Total	100.00	



## V. DISCUSSIONS

Following points are summarized based on the Examination done.

Chemical composition of samples conforms to the requirements of both B637 Gr N07080 and Nimonic 80A.

1. Microstructural examination highlights following key observations:
  - a. Specimen in Heat treatment cycle I condition showed nickel solid solution grains with twins having second phase precipitations. Presence of continuous carbides was also observed at the grain boundaries.[1,5]
  - b. Specimen with Heat treatment cycle II given does not allow the intermittent cooling at room temperature between solution annealing and aging treatments. That will have less chances of carbide formation.
2. Comparison between the microstructural properties of heat treatment cycle I specimen with Heat treatment cycle II specimen showed considerable difference in carbide precipitation pattern. The Heat treatment cycle II specimen showed that the carbides could be dissolved to such an extent that continuous carbide network at grain boundaries is disappeared. From the automated software it is observed that the grain size is in agreement with original as received structure. [1,2]
3. Scanning electron microscopy conducted on specimens with Heat treatment cycle I and Heat treatment cycle II reveals that gamma prime precipitates have further refined in Heat treatment cycle II along with formation of discrete grain boundary carbide precipitates.[1,2,5]
4. EDS analysis revealed that grain boundary consist of different carbides and gamma precipitates. The stray bright particles within the grains are found to be rich in chromium and carbon. The austenite matrix indicated Ni<sub>3</sub>Ti / Ni<sub>3</sub>Al precipitates along with nickel-chromium solid solution of alloy.

### **Based on the study, following outcomes are drawn:**

Continuous carbide precipitation was observed in Heat treatment cycle I specimen. This caused a concerned as, such microstructure can lead to decreasing of creep properties in long run [1,10,11]. Also, it was observed that Heat treatment cycle II specimen showed that carbides could be dissolved to such an extent that continuous carbide network at grain boundaries is disappeared and thus helps in improving long term creep properties.

The solution annealing treatment provides dissolving carbides followed by aging treatment that precipitates gamma prime and precipitates carbides at grain and twin boundaries. During the process of Heat treatment cycle I, intermediate air cooling between solution annealing and aging treatment however, favours higher density of grain boundary carbide precipitation. Moreover, Heat treatment cycle II was experimented by transferring the specimen from 1100°C to 850°C to avoid direct cooling from the temperature range below 850°C [1,2,5]. The result showed that most desirable discrete – globular carbides could be precipitated at grain boundary while significantly less carbide density is observed in the matrix.

The result of Heat treatment cycle I and Heat treatment cycle II specimen are thereafter compared through microstructural examinations and scanning electron microscopy. Microstructural examination revealed discrete globular chromium carbide particles and absence of continuous carbide network in Heat treatment cycle II specimen. The corrected carbide dissolution is thereby validated through above experimented heat treatment procedure.

## VI. CONCLUSION

### **Following are the conclusions drawn from the above study:**

1. The Microstructure and SEM analysis showed that the Heat treatment cycle I specimen showed Precipitation of carbide layers at the grain boundaries, which degrades the creep rupture properties, was attributed to the inherent instability of microstructures not properly heat treated to produce a distribution of coarse distinct grain boundary carbides in Nimonic 80A material.

2. The Microstructure and SEM analysis showed that the Heat treatment cycle II specimen showed non continuous carbide network, thus Heat treatment cycle II procedure is more suited to applications where long-term stability, together with the associated creep rupture ductility, are required in NIMONIC 80A material.

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