



# Extraordinary effect of the $\delta$ phase on the electrically-assisted deformation responses of a Ni-based superalloy



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## ARTICLE INFO

### Keywords:

Ni-based superalloy  
Electrically-assisted tension  
Flow stress  
Dislocation-free ring  
Local Joule heating effect

## ABSTRACT

The effect of the  $\delta$  phase ( $\text{Ni}_3\text{Nb}$ ) on the electrically-assisted (EA) deformation responses, particularly stress, elongation and dislocation distribution, of the Ni-based superalloy is still unknown up to now, though it is clear as nonnegligible on the quasi-static and hot deformation processes. Therefore, the effect of the  $\delta$  phase during the electrically-assisted tension were experimentally investigated and analyzed with comparison to that in the hot tension process. Generally, the  $\delta$  phase existing in the Ni-based superalloy always causes the reduction of flow softening and elongation during cold and the initial stage of hot tension processes. What is distinct, the  $\delta$  phase promotes the flow softening effect and elongation during the EA tension. In addition, the dislocation-free ring around the  $\delta$  phase was firstly observed during the EA tension. The mechanism of these macro and micro extraordinary phenomena is concluded as that the  $\delta$  phase causes serious lattice distortion and dislocation pile-up around itself, which leads to an extremely high temperature due to the local Joule heating effect. The faster elimination of dislocation pile-up in the local area because of the local higher temperature results in the dislocation-free ring phenomenon and the significant improvement of flow softening effect and elongation.

## 1. Introduction

Ni-based superalloy has been widely used in the aeronautical, nuclear, chemical, and marine applications, due to its excellent mechanical, corrosion and oxidation resistances at high temperature [1–3]. However, its formability is seriously limited by the high deformation resistance at room temperature. Therefore, hot working is generally used for forming Ni-based superalloy components. The microstructure and mechanical properties of the superalloy is significantly affected by the deformation characteristics such as forming temperature and strain rate etc. [4,5]. Much work has been carried out to understand how deformation characteristics influence the microstructural evolution and the mechanical properties of Ni-based superalloy. Tian et al. [6] investigated the recrystallization mechanism of a single crystal Ni-based superalloy with different crystallographic orientations and found that  $\langle 111 \rangle$  oriented sample exhibited more surface recrystallization compare to  $\langle 011 \rangle$  oriented sample. Lin et al. [7,8] investigated the microstructural evolution of a Ni-based superalloy during hot deformation with different parameters and found that the effects of deformation degree, strain rate and deformation temperature on dynamic recrystallization process are significant. All the aforementioned work demonstrated that the competition between work hardening from

dislocation pile-up and flow softening from dislocation elimination is a major factor which determines the mechanical behaviors of the material during the hot working.

Ni-based superalloy is a precipitation strengthened alloy, and the  $\gamma'$  phase ( $\text{Ni}_3\text{Nb}$ ) is the main strengthening phase. The presence of the second phase in the alloy plays a major role in determining the dislocation evolution by generating dislocation pile-ups. However, the metastable  $\gamma'$  phase can always transform to the equilibrium  $\delta$  phase ( $\text{Ni}_3\text{Nb}$ ) at the certain temperatures [9]. The existence of  $\delta$  phase can significant change the dislocation glide velocity and hence affect the mechanical response such as flow stress and strain rate sensitivity [10–13]. It can also stimulate the process of dynamic recrystallization [14,15] and affect the peak flow stress, the corresponding strain and apparent activation energy [13] during the hot working. Furthermore, the initial  $\delta$  phase content has been reported to affect the macroscopic stress response [16].

Electrically-assisted manufacturing (EAM) is one of the novel and efficient manufacturing technologies to improve productivity and quality which has attracted increasing attention recently. Decrease in flow stress, springback and increase in elongation can be achieved by applying the pulse current to a metal or an alloy [17]. The influences of pulse electric current on the mechanical behaviors have been widely

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<https://doi.org/10.1016/j.matchar.2018.08.018>

Received 27 June 2018; Received in revised form 10 August 2018; Accepted 10 August 2018

Available online 11 August 2018

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reported on different material systems [18–21]. The results demonstrated that the reduction in flow stress under given processing parameters is material dependent. On the other hand, the influence of electric current on flow stress reduction also depends on the strain rate which results from the Joule heating effect [22]. The elongation of AA5052 [23] and AA5754 [24] alloys during electrically-assisted (EA) tension can increase up to 100% and 400% respectively under the appropriately pulse current density and pulse frequency. Not only the mechanical behaviors, but also the microstructure of the material can be improved by applying pulse current. The electropulsing treatment can significantly promote the recrystallization behavior of the alloy when temperature is much lower than the recrystallization temperature [22,25,26]. The investigation of the dislocation structure after EA tension revealed that the dislocations may disappear under the pulse current by the replacement of subgrain or the formation of tangled dislocations [22,27].

The pulse electric current during EAM has been demonstrated that play an important role in determining the mechanical response of materials from the microstructural length scale. Similarly, the grain size distribution [28,29] and dislocation density [30] also show significant effects on the plasticity during EA manufacturing process. However, the effect of second phase on the EA process are barely reported, even though the presence of the second phase is believed to be a non-negligible factor of hot working. Recently, the plastic deformation mechanism of GH4169 alloy under electric-pulse current at high temperature were recently reported by An et al. [31,32], but the effects of  $\delta$  phase in Ni-based superalloy remains elusive.

The purpose of this study is therefore to investigate the role of second phase and the effect of different initial microstructures on the mechanical behaviors and dislocation evolution during the EA tension and hot tension. A systematic study consists of a series of uniaxial tensile tests over wide ranges of deformation parameters. In what follows, firstly, the effect of the EA characteristics such as peak current density in EA tension and furnace temperature in hot tension are detailed analyzed. Then the dislocation evolution of the region without  $\delta$  phase and the region around the  $\delta$  phase during the EA tension and hot tension are compared using transmission electron microscopy (TEM). At the end the special effect of the  $\delta$  phase on the mechanical behaviors and dislocation evolution during the EA tension and the mechanism of it are presented and discussed.

## 2. Experimental Procedure

### 2.1. Material

The material employed in this study is a Ni-based superalloy (Inconel 718) sheet with the thickness of 1 mm. The detailed chemical composition of this superalloy is shown in Table 1. Two types of different specimens were prepared in this study in order to investigate the effect of  $\delta$  phase: (a) the as-received specimens and (b) the aging treatment specimens. The later comes from the aging treatment of the as-received specimens at a temperature of 900 °C for 24 h followed by water quenching.

The initial grain size distributions were assessed by electron back-scattered diffraction (EBSD) technique while the morphology of  $\delta$  phase was observed by scanning electron microscope (SEM). The specimens for EBSD and SEM analysis were fabricated by wire-cut machine along the transverse direction (TD) of the specimens. Then, the sections of the

**Table 1**

The chemical composition of the Ni-based superalloy in the present work.

Material	Chemical composition (wt%)								
	Ni	Cr	Nb	Mo	Ti	Co	Al	Si	Fe
Inconel 718	53	17.79	5.11	3.13	0.94	0.54	0.46	0.34	Balanced

specimens for EBSD analysis were polished by mechanical polishing machine followed by electro-polishing in a solution of 30 ml HClO<sub>4</sub> and 270 ml CH<sub>3</sub>CH<sub>2</sub>OH. TSL OIM Analysis software was used for the EBSD data acquisition and post-processing. The sections for SEM analysis were also polished by mechanical polishing machine and then chemically etched in a solution of HCl (100 ml) + CH<sub>3</sub>CH<sub>2</sub>OH (100 ml) + CuCl<sub>2</sub> (5 g) at room temperature.

The obtained microstructures and grain morphology are shown in Fig. 1. It can be found that the initial microstructures of both specimens are similar and mainly composed of equiaxed grains and a small amount of annealing twins. The average grain size is 51.13 mm and 53.81 mm, respectively. It indicates that the aging treatment almost has almost no influence on grain size. The distribution of  $\delta$  phase is shown in Fig. 2(b) with comparison with that of the as-received specimen (Fig. 2a). However, the  $\delta$  phase is introduced to the specimen after the heat treatment shown in Fig. 2. The needle-like  $\delta$  phases is found precipitate heavily after aging treatment and the content of initial  $\delta$  phases can be statistically evaluated as 22.76%.

Additionally, TEM was employed to study the effect of  $\delta$  phases on the dislocation structure. To prepare the sample for TEM, thin foils with a diameter of 3 mm were punched out at the geometry center of the specimens after being ground to a thickness of 0.03 mm, and then were electropolished in a corrosive solution of CH<sub>3</sub>CH<sub>2</sub>OH and HClO<sub>4</sub> (9:1 in volume fraction).

### 2.2. Test Approaches

To carry out the EA and hot tension tests, the profiles of the specimen are prepared, respectively, as illustrated in Fig. 3(a) and (b). In order to apply pulse current loading, the blue areas are designed at the two ends of the sample for EA tension as shown in Fig. 3(a), while its gauge length keeps consistent with that of the specimen for hot tension (Fig. 3b) for direct comparison. The tension tests were conducted using a CMT4000 testing machine. A pulse power supply (GMCW-F20V-100A) was used for the EA tension tests and the detailed parameters of which are listed in Table 2. A furnace was employed for hot tension tests.

During the EA tension, a set of rubber insulators were inserted between the gripper and the machine to ensure excellent insulation. An infrared camera and a current detector were used to record the instant temperature and current value, respectively.

## 3. Results

### 3.1. Effects of $\delta$ Phase on Stress-strain Responses

The samples from the as-received and aging treatment materials are subjected to the EA and hot tension tests to investigate the effects of  $\delta$  phase on stress-strain responses. In order to make the results between these two types of tests comparable, the temperature history of the specimen surface during the EA tension is first measured, and then the temperature range for hot tension tests are set to be identical to the maximum temperature reached in the EA tension. It worth to point out that a slight difference between the two tests as that the temperature increases from room temperature to its maximum gradually because of the Joule heat during the EA tension tests.

The comparison of the stress-strain responses is shown in Fig. 4, where Fig. 4(a) and (b) are from the as-received specimens and Fig. 4(c) and (d) are from the aging treated ones. Fig. 4(a) and (c) are the results from the EA tension and Fig. 4(b) and (d) are the results from the hot tension. It can be found that, with the as-received material, the flow stress exhibits the similar tendency during both the EA tension tests and the hot tension tests: decreasing significantly with the increasing peak current density or the increasing temperature, as shown in Fig. 4(a) and (b). However, the flow stress decreases more significantly during the EA tension when the final maximum temperature is close to the furnace

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