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EFFECTS OF ETA PHASE ON CREEP PERFORMANCE OF THE NICKEL-BASE SUPERALLOY 263

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ABSTRACT

In wrought nickel-base alloys used at elevated temperatures for extended periods of time, it is commonly observed that unwanted phases may nucleate and grow. One such phase is the eta phase, based on Ni_3Ti, which is a plate-shaped precipitate that nucleates at the grain boundaries and grows at the expense of the strengthening gamma prime phase. In order to study the effects of eta phase on creep performance, Alloy 263 was modified to contain 3 different microstructures: standard (contains gamma prime); aged (contains gamma prime and eta); and modified (contains only eta and no gamma prime). These microstructures were then creep tested in the range of 973-1123 K (700-850°C). An extensive test matrix revealed that the eta-only modified alloy had creep rupture strengths within 10% of the standard alloy even though this alloy had no strengthening gamma prime precipitates. It also exhibited superior creep ductility. A preliminary test matrix on the aged material containing eta and gamma prime prior to the creep tests revealed that the performance of this microstructure was generally between that of the standard alloy (best) and the eta-only alloy (worst). The aged material exhibited far superior creep ductility. These results suggest that the presence of the eta phase may not be deleterious to creep ductility, and in fact, may enhance it.

INTRODUCTION

Wrought nickel-base alloys such as INCONEL® 740/740H and NIMONIC 263 are being considered for high-temperature, high-efficiency electric power generation facilities. With few exceptions, these alloys are strengthened by the gamma prime phase, designated γ', and based on the $Ni_3Al/L1_2$ structure. During extended times at operating temperatures, other phases can form, and many of these phases are considered to be detrimental to performance. One such phase that forms in these types of alloys is the eta phase, designated η, and based on the $Ni_3Ti/D0_{24}$ structure [1]. The η phase is known to form at the expense of the γ' strengthening phase at long service times. Examples of this phase formation are shown in Figure 1 from a study by Unocic et al. in IN740 [2].

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It has been speculated that this development of η phase might be deleterious to creep performance. Not only does the η phase reduce the volume fraction of the γ' strengthening phase, but it also occurs at grain boundaries in sharp shapes, perhaps leading to enhanced cavitation and crack initiation.

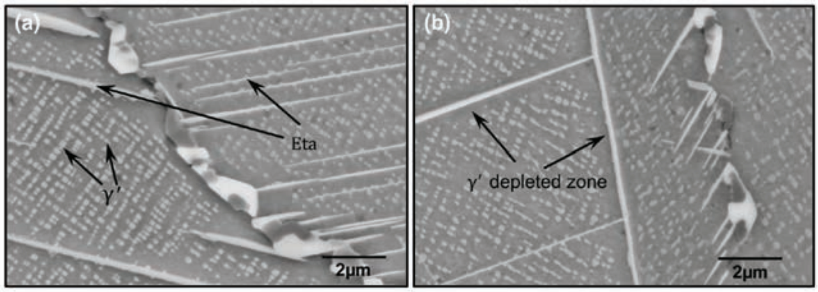


Figure 1. Development of η phase plates in IN740 after 23,000 hours at 1023 K (750°C) [2]

However, in the available studies of the effects of η phase on performance in the literature, there is no broad agreement that η phase is detrimental, as summarized in Table 1.

Table 1. Summary of literature observations of η phase on performance of typical alloys

Alloy [Reference]	**Temperatures, K**	**Observations**
N263 [3]	1073	Reduced creep ductility
N263 [4]	1100	Detrimental to strength and ductility
IN740 [5]	1023-1123	Reduced impact toughness
IN740 [6]	1090	Reduced strength, limited ductility
IN740 [7]	1023-1123	Reduced creep ductility above 7% η
IN740 [1]	*1023*	*Not detrimental to creep*
IN740 [2]	*1023*	*Not detrimental to creep*

The studies summarized in Table 1 varied significantly in stress levels and durations of the tests, as well as in the η volume fraction. Furthermore, none of these studies incorporated detailed evaluation of the effects of η phase formation on constitutive behavior during creep. The present study was undertaken to clarify the effects of η phase formation on the creep performance, and in particular the constitutive behavior, of these alloys.

PROJECT GOALS

In all previous studies, the η phase evolved during the creep tests after long service times at elevated temperatures. Other microstructural features also evolved during these creep tests that may have confused the interpretation of the η phase effects. In order to clarify the effects of the η phase on creep performance of Alloy 263, three different microstructures were produced by alloy design and heat treatment prior to creep testing:

1. “Standard” Alloy 263 containing only γ'.
2. “Aged” Alloy 263 containing both γ' and η.
3. An experimental alloy similar to Alloy 263 which contains only η and no γ'.

Thus, three different microstructures containing 3 different distributions of γ' and η were produced and creep tested. The project is in its intermediate stage, so some of the creep data is already available but many more tests are currently underway or planned.

MATERIALS AND MICROSTRUCTURES

The compositions of Alloy 263 and the experimental "Alloy 20" are given in Table 2.

Table 2. Chemical compositions of the alloys (weight pct)

	Ni	Co	Cr	Al	Fe	Mo	Mn	Si	Ti	Nb	W	Ta	V	C
263	Bal	20	20	0.6	0.7	6	0.6	0.4	2	0	0	0	0	0.06
20	Bal	21	21	0.14	0.5	0	0.4	0.2	2.8	2	2	1	0.85	0.07

Alloy 20 was designed in a previous study to be similar in composition to Alloy 263, but to contain only η and no γ' [8,9]. Its microstructure after aging at 1123 K is shown in the TEM and SEM images in Figure 2, where only plate-like η phase is present in a Widmanstätten microstructure [8,9].

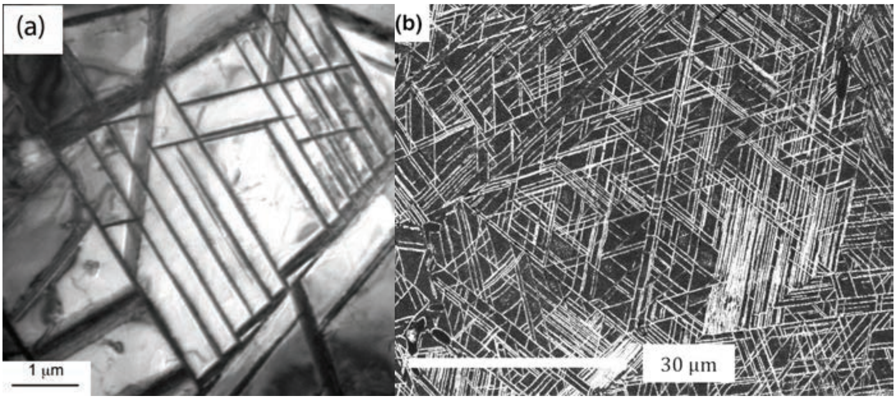


Figure 2: Microstructures of the experimental Alloy 20 after aging at 1123 K, designed using computational thermodynamics to contain only η and no γ' [8,9]. η plates in a Widmanstätten microstructure are the dominant features. (a) TEM micrograph of Alloy 20. (b) SEM micrograph of an alloy with a very similar composition.

The "standard" Alloy 263 is a polycrystalline, wrought alloy containing approximately 15% γ', along with grain boundary carbides. The γ' in this alloy is spherical, and approximately 40 nm in diameter after solution treating, quenching and aging.

In order to generate microstructures containing η and γ' prior to creep testing, aging experiments were conducted for extended times at temperatures between 1023 K (750^{o}C) and 1173 K (900^{o}C). These temperatures were chosen based on an Isothermal Transformation diagram for η and γ' precipitation that was proposed for Alloy 263 by Zhao et al. [10]. The results of these experiments were as follows:

1. There was very little η precipitation at 1023 K (750^{o}C) at times up to 7,000 hours.
2. 1173 K (900^{o}C) was at or near the η and γ' solvus temperatures, as very little η or γ' was present upon quenching from 1173 K.
3. Useful microstructures containing η and γ' could be produced at 1073 K (800^{o}C) and 1123 K (850^{o}C), as explained below.

Figure 3 shows an SEM micrograph of an Alloy 263 specimen that had been aged at 1123 K (850°C) for 1,000 hours. This micrograph illustrates the polycrystalline structure of the alloy, along with the spherical γ' precipitates in the centers of the grains, and plate-shaped η precipitates near the grain boundaries and protruding into the grains. Note the similarities between this structure and that shown in Figure 1, developed in a similar alloy during creep after 23,000 hours at 1023 K.

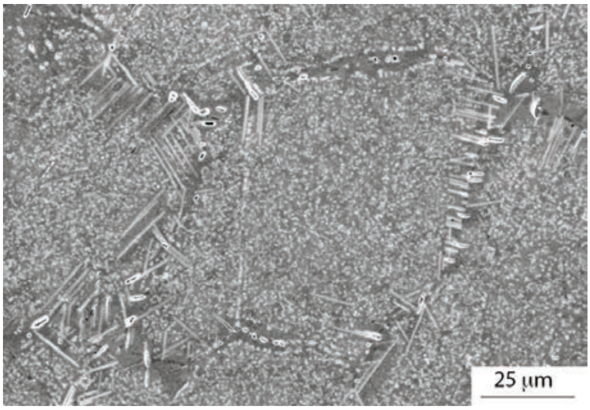
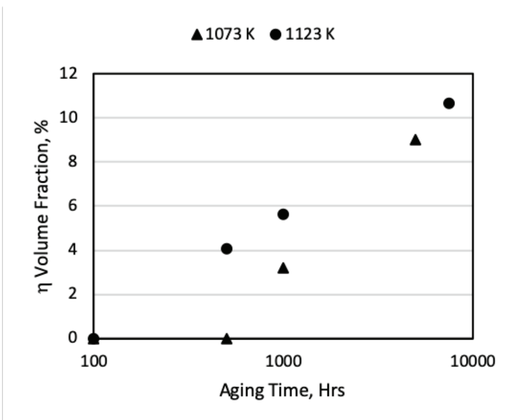
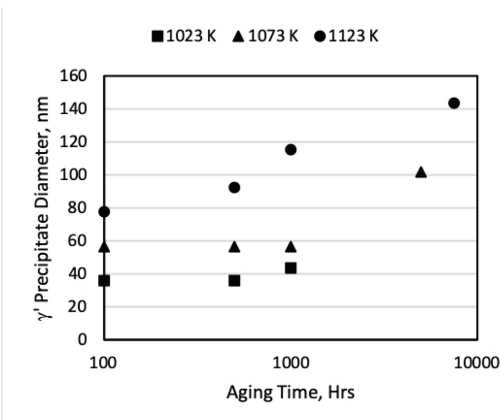


Figure 3: SEM micrograph of Alloy 263 after aging at 1123 K (850ºC) for 1,000 hours. Note presence of plate-shaped η precipitates *near grain boundaries.*

In addition to the precipitation of η, heat treatment of Alloy 263 at these times and temperatures coarsens the γ', and some of the γ' near the grain boundaries dissolves at the expense of the of η growth. Figure 4 shows the evolution of η volume fraction and γ' precipitate diameter during aging at these temperatures. The goal of the heat treatments was to obtain a microstructure with a substantial volume fraction of η while limiting the γ' coarsening. Unsurprisingly, these goals turned out to be mutually exclusive; the compromise choice for the (η+γ') microstructure is the one shown in Figure 3, aged at 1123 K for 1,000 hours. This microstructure contains about 6% η by volume, and the remaining γ' precipitates have coarsened to a diameter of around 115 nm.



(a)



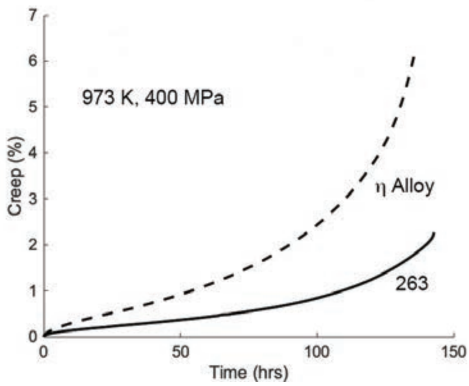
(b)

Figure 4. Microstructural evolution of Alloy 263 with aging times. (a) Volume fraction of η *phase. (b) Diameter of* γ' *precipitates.*

RESULTS – CREEP PERFORMANCE OF THE η-ONLY ALLOY

Initial results and a detailed discussion of the research program are available in [9]. Creep tests were performed on the experimental alloy that contains only η phase, at a range of stresses, and at temperatures of 973, 1023 and 1073 K (700, 750 and 800°C.) Since the publication in [9], the creep test matrix was finished and the complete results are discussed below.

Figure 5(a) shows typical creep curves for both the experimental alloy and a standard Alloy 263 specimen. Figure 5(b) shows a Larson-Miller plot of the data for the experimental η alloy, along with a design curve of average data for 263 from the material producers [11-13]. Alloy 263 outperforms the experimental η alloy by approximately 10% on this basis (but slightly less at high rupture strengths.). It is rather interesting that the experimental η alloy, which contains no strengthening γ' precipitates, actually performed this well. Also, the creep ductility of the experimental η alloy was always superior to that of the standard Alloy 263. TEM studies of the deformation mechanisms in the experimental η alloy are presented in [9], and a more detailed report is currently being prepared.



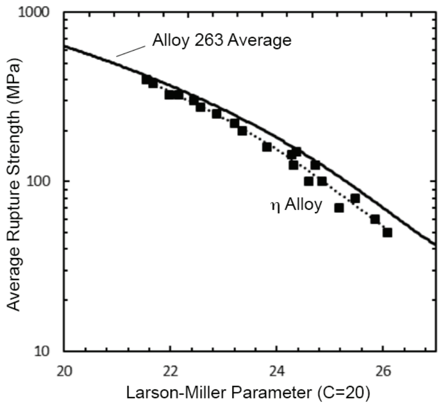


Figure 5: (a) Typical creep curves for the experimental alloy containing only η phase and a standard Alloy 263 microstructure. (b) Larson-Miller plot showing rupture strength performance of the experimental η alloy vs standard Alloy 263 averages [11-13].

RESULTS – CREEP PERFORMANCE OF THE AGED ALLOY 263

Preliminary results from a significant test matrix are available at this time and are discussed below. Figure 6 shows creep curves for all three microstructures at temperatures of 1073, 1123 and 1173 K (700, 750 and 800°C). In some cases, results at slightly different stresses are all that is currently available, and they are labeled as such on the graphs. The following observations may be made:

1. With the exception of the highest stress level at the lowest temperature (Fig. 6, top-right), the creep behavior of the aged alloy containing η and γ' lies between the standard 263 (best) and the η-only alloy (worst).
2. The creep ductility of the aged alloy containing $\eta + \gamma'$ is far superior to the standard 263 and is also better than the η-only alloy.

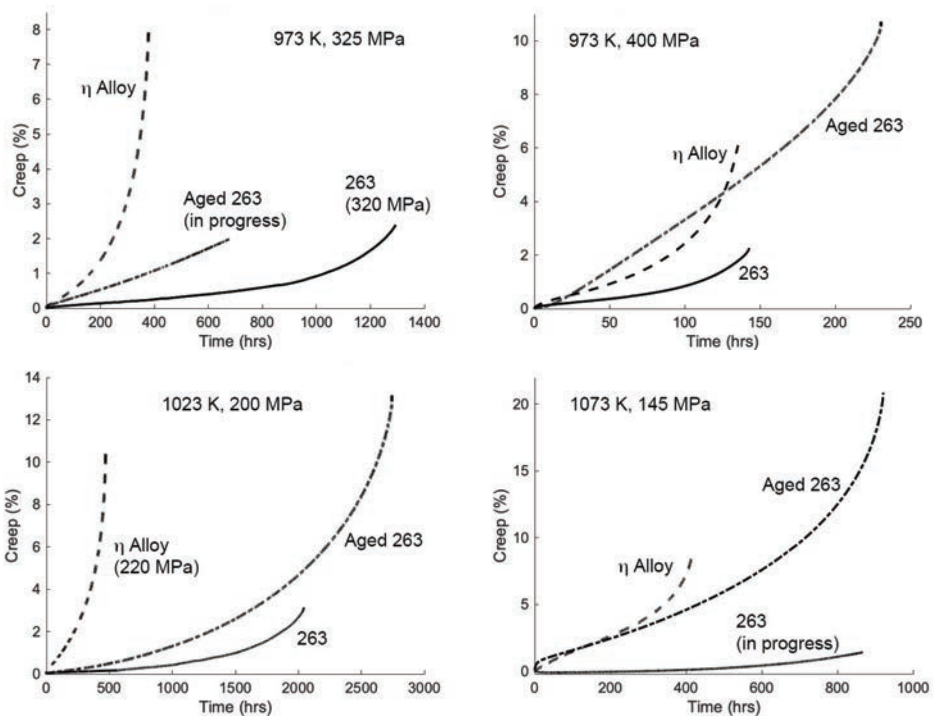


Figure 6: Creep curves. The Aged 263 alloy contains η and coarsened γ'.

In addition to finishing the test matrix, significant research is needed to understand the creep behavior presented here. The creep rates are higher in the aged alloy than in the standard alloy, and the creep ductilities are higher. There are several possible factors (or combinations of factors) that may cause this:

1. Presence of η.
2. Increased γ' precipitate diameter.
3. Reduced γ' volume fraction.
4. γ' depleted zones near grain boundaries and η.

TEM studies of deformation mechanisms, along with microstructure-informed constitutive modeling studies are underway to address these issues. One observation that might be evident from Figure 6 is that the presence of η may not deleterious to creep ductility. Many studies have implied that the plate-shaped precipitates in the grain boundaries would accentuate cavitation, cause stress concentrations, and reduce creep life for these reasons. This data appears to show the opposite effect.

CONCLUSIONS

Alloy 263 can be modified by chemistry and heat treatment to contain three different microstructures: γ'-only (standard alloy); η-only (modified chemistry and heat treatment); and η+γ' (aged standard alloy). A substantial creep test matrix on the η-only alloy determined that this alloy, containing no γ', has rupture strengths within 10% of the standard alloy, and superior creep ductility. Preliminary creep results on the aged material containing η+γ' indicated that its creep performance falls between the standard alloy and the η-only alloy, but its creep ductility is far superior to the standard Alloy 263 and is also better than the η-only alloy. This suggests that η precipitation may actually be beneficial to creep ductility, and not detrimental as has often been proposed.

ACKNOWLEDGMENTS

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