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Characterization of strain induced precipitation in Inconel 718 superalloy

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Abstract. Inconel 718 is a Ni-Cr-Fe superalloy which presents excellent mechanical properties at high temperatures, as well as good corrosion resistance and weldability. These characteristics can be optimized with an appropriate control of microstructural features such as grain size and precipitation. Precipitates of different nature can form in these alloys, *i.e.* γ'' (a metastable metallic compound Ni_3Nb), γ' (Ni_3(Ti, Al), carbides and/or δ phase (intermetallic Ni_3Nb). Aging treatments are usually designed to obtain the precipitation required in order to optimize mechanical properties. However, precipitation can also appear induced by deformation and therefore interfere with hot

forming operations, such as forging. In particular, the formation of δ phase is a concern regarding[j1] deformation processes, because it forms at the highest temperatures. In this work, stress relaxation tests were carried out in order to characterize the precipitation kinetics of Inconel 718 at temperatures ranging from 950°C to 800°C. Moreover, the effect of deformation on precipitation was also assessed by applying different degrees of deformation, *i.e.* 0.005, 0.1 and 0.2, prior to stress relaxation. The analysis of the relaxation curves, together with a proper characterization of precipitation by Scanning Electron Microscopy, was the basis for obtaining precipitation-time-temperature (PTT) diagrams, as well as the effect of deformation on precipitation kinetics.

1. Introduction

Inconel 718 superalloy is commonly used for its outstanding corrosion and mechanical properties at high temperatures, *i.e.* it can be used at temperatures up to 650ºC, being one of its main applications in gas turbine parts. These properties rely on the formation of γ'' (Ni_3Nb) phase, with a body centered tetragonal structure coherent with the matrix, which is the primary strengthening precipitate in Nb containing nickel-iron superalloys [**Error! No s'ha trobat l'origen de la referència.**]. Other precipitates, such as γ' phase $Ni_3(Ti, Al)$ can form simultaneously with γ'' at temperatures ranging between 550 ºC and 660ºC and long aging times [**Error! No s'ha trobat l'origen de la referència.**-3]. These γ' precipitates are coherent with the matrix and they contribute to strengthening, but to a lesser degree than γ''. A distinct characteristic of the precipitation taking place in Ni-based superalloys containing Nb and Fe, such as Inconel 718, is their susceptibility to the formation of δ phase (Ni_3Nb), which can be considered the equilibrium phase of γ'' [4] . This phase, which usually exhibits plate morphology [ref][j2], has an orthorhombic structure incoherent with the matrix, and therefore does not significantly contribute to strengthening.

Even thought precipitation is usually considered from the point of view of strengthening, and most of the investigations have focused on aging treatment assessment [5, 6], precipitation can also appear induced by deformation and therefore interfere with hot forming operations, such as forging [7]. In particular, the formation of δ phase is the main concern regarding forming processes, because this phase exhibits its fastest precipitation kinetics around 900ºC and can appear at temperatures as high as 1000ºC, which is its solvus temperature. The presence of δ-phase can be detrimental because it depletes the formation of the γ'' and is associated with an increase in the susceptibility to hot cracking. However, under certain circumstances, i.e. during solution treatments, δ phase can be helpful in controlling grain growth and has been shown to provide resistance to grain boundary creep fracture [6].

The characteristics of hot deformation of Inconel 718 have been reported by several authors [7-10] and even the behavior at high temperatures when δ precipitates are present in the microstructure, have been evaluated [10-12]. However, the effect of deformation on inducing precipitation has not yet been studied.

In this work, the precipitation kinetics of Inconel 718 has been evaluated from the point of view of its effect on processing. With For this purpose, stress relaxation tests were performed at different temperatures [13]. It is known that deformation can accelerate precipitation kinetics. The effect of deformation on precipitation kinetics has also been evaluated by applying different levels of strain prior to relaxation. Precipitation-temperature-time (PTT) curves were derived from stress relaxation curves, and an accurate metallographic observation work was realized done in order to identify the species forming at different testing conditions.

2. Experimental method

The material used for the experimentation was an Inconel 718 superalloy with the composition listed in table 1. This material belongs to the nickel-chromium-iron age-hardenable austenitic superalloys family, and it contains other elements such as Nb, Mo, Al and Ti, which combine to offer high corrosion resistance, high strength and excellent creep resistance up to 650ºC. The as-received material had been annealed at 980ºC for 1h and then water quenched. The hardness associated with this condition was 292HV.

In order to homogenize the microstructure and composition, the material was subjected to reheating treatments at 1100ºC for 1h in a tubular furnace. Fig. 1 shows FE-SEM images of the as-received microstructure (Fig. 1a) and the microstructure after reheating (Fig. 1b). In general, the precipitation of δ phase, which was prominent in the as-received microstructure, disappears after the abovementioned solution treatment at a temperature above the δ solvus temperature. However, other Nb,Ti carbides could not be dissolved. The solution heat treatment promoted and average grain size of 74μm. This was the initial microstructure for the subsequent mechanical tests.

After heat treatment, cylindrical samples 10mm in length and 5mm in diameter were machined for mechanical testing in a Bahr dilatometer DIL805A/D. The thermomechanical schedule followed by the samples is shown in Fig. 2. During testing, the samples were heated up to 1100ºC and they remained at this temperature for 5 min until the temperature was homogeneous throughout the sample. After reheating, the samples were cooled down to the testing temperature which ranged from 800ºC to 950ºC. After a short stabilization period of 10s, the samples were deformed to different strain levels (0.005, 0.1 and 0.2) and then relaxed, *i.e.* deformation was kept constant and the variation of the stress with the time was recorded [14]. The different deformation levels were selected to evaluate the effect of deformation on precipitation characteristics. The smallest deformation level was 0.005, which was within the elastic

range of deformation for all chosen temperatures. The relaxation curves under these conditions will givegave information about the kinetics of precipitation when there is no plastic deformation and, thus, generation of dislocations involved. On the other hand, plastic deformation was introduced for 0.1 and 0.2 deformation conditions and the initial dislocation density was higher after 0.2 deformation.[j3]

After stress relaxation, samples were quenched to retain the microstructure for further metallographic observation. Moreover, additional samples were quenched at intermediate relaxation times to assess the progress of precipitation at the different testing temperatures. Conventional sample preparation was carried out before etching the samples with Kalling's reagent and subsequent metallographic characterization was performed in a Field Emission Scanning Electron Microscope (FE-SEM) JEOL JSM-7001.

3. Results

3.1. Precipitation characteristics of non-plastically deformed specimens

Relaxation curves

Stress relaxation curves after 0.005 deformation can be seen in Fig. 3 at different temperatures. In general, the curves exhibit a hump in the stress vs time curve, when relaxation starts. After reaching a maximum value, the stress drops lineally with the log of time until the relaxation is arrested and the relaxation rate is reduced. Usually, it is considered that an increase in the relaxation rates is associated with softening mechanisms, whereas a decrease in the relaxation rates is associated with strengthening mechanisms. In the curves, the inflection points, *i.e.* changes in the relaxation rates, are indicated with an arrow. Arrows with a continuous line indicate a decrease in the

relaxation rate and arrows with a pointed line indicate an increase in the relaxation rate. Given the nature of the material being tested, the strengthening mechanism responsible for the decreases in the relaxation rates is ~~most likely~~ precipitation, i.e. precipitates interact with dislocations motion and inhibit relaxation. On the other hand, ~~when there is~~ a decrease in the relaxation rate~~, this could~~ can be associated with softening mechanisms, such as recrystallization or recovery, or to the end of precipitation and coarsening of the formerly formed precipitates.

Characterization of the evolution of the microstructure

In order to evaluate the effect of the temperature on the precipitation taking place for Inconel 718 when no deformation is involved, several samples were characterized at different relaxation temperatures and times. At the lowest testing temperature of 800ºC, the microoestructures of samples quenched after 60s and 3600s relaxation times are compared in Fig. 4. The first sample was quenched after 60s because this relaxation time corresponds to the maximum stress value. Since the humps at the beginning of the relaxation curves seem to indicate a strengthening of the material, it was checked whether it could be related to precipitation. According to Fig. 4a, there is no precipitation, so the origin of the hump is not related to this phenomenon. The nature of the deformation applied to the samples, within the elastic regime, could be the responsible of this ~~king of~~ behavior in non-plastically deformed samples. [j4]

Fig. 4b shows that 800ºC is a temperature at which precipitation can take place extensively and the precipitation start and finish times would be the ones indicated in the corresponding relaxation curve in Fig. 4b. The precipitates ~~which form~~ appear mainly in the matrix and are oriented with respect to the crystallographic orientation. Given their disk-like shape, they can be identified as γ''. [ref][j5]

At higher temperatures, the characteristics of precipitation change. ~~Precipitation~~ Precipitates at 850ºC (Fig. 5) appear~~s~~ mainly at grain boundaries and near ~~big~~ large Nb(C,N) particles~~precipitates~~. The shape of these precipitates is plate-like, so they can be identified as δ phase [ref]. At higher relaxation temperatures, no significant precipitation ~~could be~~was detected. ~~In Fig. 6~~ ~~T~~the presence of some δ phase at grain boundaries is evident at 900ºC after 900s, as shown in Fig. 6. H~~h~~owever, this precipitation is very weak and at 950ºC it was not detectable, so the changes in the slope of the relaxation curves corresponds to the natural relaxation behavior of the material.

PTT curve

The PTT curves derived from the stress relaxation curves and evaluation of the microstructures is represented in Fig. 7. These curves indicate approximately the temperature range and times and which each type of precipitates form on cooling from above δ solvus, which is around 1000ºC [15], under isothermal conditions. These results are in agreement with similar diagrams published in the literature [7,15].

3.2. Precipitation characteristics of 0.1 and 0.2 deformed samples

Relaxation curves

The relaxation curves of the samples deformed 0.1 and 0.2 are represented in Fig. 8. As previously for the non-plastically deformed samples, the solid arrows in the graphs indicate the inflexion points at which the relaxation is arrested and dashed arrows indicate the inflexion points at which relaxation is accelerated.

Relaxation curves exhibit similar characteristics for the 0.1 and 0.2 deformed samples. At low temperatures, *i.e.* 800ºC and 825ºC, the curves show several inflections during the relaxation time. The first arrest takes places around 10s and it could be related to the presence of strain induced precipitates. After this period, relaxation proceeds at higher rates, related to the activation of restoration processes, typically recovery or

recrystallization. After this, there is another arrest of the relaxation rates, this time more pronounced, which indicate that the precipitates which form at this point, *i.e.* around 200s, have a bigger strengthening capability.

At 850ºC, and for the sample deformed 0.2, there is an arrest of the relaxation rate which is followed by another decrease of the relaxation rate, which indicates that after the beginning of a first precipitation, a second precipitation phenomenon appears at lower relaxation times, with no time for restoration in between. For the 0.1 deformed samples, the dislocation density is probably not sufficient to promote precipitation at short relaxation times.

At 875ºC and higher temperatures, there is only one precipitation phenomenon which takes place around 100s.

Characterization of the evolution of the microstructure

~~The evolution of the microstructure was analyzed, paying special attention to the precipitation characteristics, for the 0.2 deformation conditions.~~ The amount of deformation is expected to have an effect on the precipitates volume fraction. However, given the similar behavior indicated by the relaxation curves between the 0.1 and 0.2 deformation conditions, only the later condition has been microestructurally [j6]analyzed ~~microestructurally~~.

Fig. 9 represents the microstructure of the samples relaxed after 0.2 deformation at 800ºC, quenched after 40s (Fig. 9a) and after 900s (Fig. 9b and 9c). After 40s, the relaxation curves indicate that a first precipitation phenomenon might have taken place. According to the Fig. 9a, this precipitation takes place at grain boundaries. In Figure 9b, for a more advanced stage of the precipitation, it can be seen that precipitation is extensive both at grain boundaries and in the matrix. Precipitation in the matrix is the responsible for the second precipitation phenomena detected by the stress relaxation

technique. The precipitates at grain boundaries are not plate-like, so they are assumed to have have probably the same nature of the precipitates which form after longer relaxation times, most likely i.e. γ'', which is the unstable form of δ. The precipitates that appear within the matrix do not show any specific shape. They could be γ'' precipitates which have not got time to evolve to a disk-like shape, or could be γ' precipitates. Usually, the formation of γ' would require long aging times [7]. However, the presence of this phase should not be disregarded when deformation is applied before aging. The determination of the exact nature of the small precipitates that appear in the matrix requires the use of more specific techniques and this was not the object of this paper. At 850ºC the amount of precipitation at grain boundaries and in the matrix is more pronounced that for lower temperatures (Fig. 10) but the nature of the precipitates is the same.

In the case of samples relaxed after 0.2 strain at 900ºC (Fig. 11 and 12), the nature of precipitation was completely different. After 600s, which is a relaxation time in the middle of precipitation start and finish precipitation times, precipitation takes place at grain and twin boundaries (Fig. 11). Precipitation is more extensive than the one found at the same temperature for longer relaxation times after no plastic deformation as shown in Fig. 6b. However, the morphology of the precipitates is similar, and given the temperature range at which they are forming and the plate-like shape, they can be identified as δ phase precipitates.

The precipitation level at the end of the relaxation at 900ºC can be observed in Fig. 12. Under such conditions, the precipitation appears throughout the whole sample, *i.e.* at grain and twin boundaries, as well as within the grains. The morphology of the precipitates which appear at grain and twin boundaries is similar to the one found after lower strains and/or relaxation times. However, other smaller disk-like precipitates

indicated by black arrows also appear in the matrix (Fig. 12c), together with other small spherical precipitates indicated by white arrows. Disk-like precipitates are all oriented in the same direction within the same grain. The shape and orientation of these precipitates indicate that they ~~could~~ must be γ'' precipitates, whereas the spherical precipitates inside the grains ~~could~~ should be γ' precipitates, which would form after short aging times under deformation conditions, or stable δ precipitates which might have formed from metastable γ'' precipitates.

PTT curves

The PTT curves generated from the stress relaxation curves in Fig. 8 and the precipitation characterization according to Fig. 9 to 12 can be seen in Fig. 13. The curves differentiate precipitation at grain boundaries and within the matrix at low temperatures and the formation of δ phase at high temperatures. The curves of the 0.1 and 0.2 deformation conditions are superimposed in the same figure. According to the results, and as expected, the precipitation kinetics are faster after the 0.2 deformation than after 0.1 ~~deformation~~. However, bigger differences are found when the precipitation kinetics of non-deformed specimens (Fig. 7) is compared to the precipitation kinetics of deformed specimens (Fig. 13).

4. Discussion

In Fig. 3 and 8 the relaxation curves after different strain levels have been compared for all testing temperatures. However, this representation is not useful to compare the curves according to the deformation applied to the samples prior to relaxation. This comparison can be seen in Fig. 14 for all testing temperatures. In this case, the relaxation curves represent the normalized stress vs the log of time, where the

normalized stress is the relaxation stress at each time divided by the stress at the beginning of the relaxation, σ_0. Since the initial stress, σ_0, is different for each initial deformation condition, the only way to evaluate the effect of deformation on relaxation curves is by comparing the normalized curves. In this case, the arrows indicating decreasing relaxation rates, i.e. strengthening mechanisms, and increasing relaxation rates, i.e. softening mechanisms, have also been indicated.

According to the curves in Fig. 14, the differences between the 0.005 deformed samples, and the 0.1 and 0.2 deformation curves are ~~also~~ evident. In elastically deformed samples (~~After~~ strain of 0.005) the stress does not drop until several seconds after the beginning of the relaxation and, for the lower relaxation temperatures, there is even an increase of the stress at the beginning of the relaxation. Traditionally, an increase on the stress during relaxation is related to a strengthening mechanism ~~which,~~i.e precipitation in the present alloy ~~given the material and experimental conditions, could be precipitation~~. However, Fig. 4a showed that no precipitation could be seen in samples quenched after relaxations times where the normalized stress was close to the maximum. The difference between 0.005 and 0.1 and 0.2 deformed samples can be attributed to the nature of deformation. In Fig. 15, it is proved that 0.005 deformation is within the plastic deformation regime, even at the highest deformation temperature of 950ºC, whereas 0.1 and 0.2 deformation implied plastic deformation. According to the fundamentals of relaxation testing [14]:

$$\varepsilon_t = \varepsilon_e + \varepsilon_p = cte \tag{1}$$

where ε_t is the total strain, ε_e is the elastic strain and ε_p is the plastic strain. During the progress of relaxation ε_t is kept constant, and therefore~~which means that~~:

$$\dot{\varepsilon}_t = \dot{\varepsilon}_e + \dot{\varepsilon}_p = 0 \tag{2}$$

Accordingly~~and therefore~~:

$$\dot{\varepsilon}_e = -\dot{\varepsilon}_p \qquad\qquad (3)$$

This means that ~~during relaxation~~ the plastic deformation increases during relaxation in the expense of the elastic deformation.

In the case of 0.005 deformation conditions, there was no initial plastic deformation. Applying the ~~so, according to~~ Hooke's law:

$$\varepsilon_t = \varepsilon_e = \frac{\sigma}{E} = 0.005 \qquad\qquad (4)$$

where σ is the stress and E is the Young's modulus.

Therefore,

$$\dot{\varepsilon}_t = \dot{\varepsilon}_e = \frac{\dot{\sigma}}{E} = 0 \qquad\qquad (5)$$

Since E is a constant, the mechanical evaluation of stress relaxation curves when only elastic deformation is applied prior to relaxation, implies that $\dot{\sigma} = 0$. In fact, this is a characteristic of the relaxation curves after 0.005 strain, which start all horizontal and some time is required before the relaxation rate changes.

As previously indicated, the increase in the stress after the beginning of relaxation for elastic deformation conditions cannot be explained in terms of precipitation. It is believed that this behavior is related to the stiffness (compliance) of the system. In fact, during the deformation of the samples the machine is also compressed. This compression of the system diminishes during relaxation implying a further compression of the sample during the first stages of relaxation and, thus, a small increase of the stresses, which is evident in the curves. This effect of the stiffness of the system becomes less pronounced as the testing temperature is increased.

In terms of the inflexion points, the samples deformed 0.005 exhibit a similar behaviour to the 0.1 and 0.2 deformed samples at high temperatures, i.e. 950ºC, 900ºC and 875ºC.

In general, precipitation starts around 100s and finishes around 1000s. However,~~,~~ it is slightly shifted towards lower times at higher applied strain~~although this phenomenon~~

~~appears slightly earlier the higher the strain applied prior to relaxatation~~, which indicates that precipitation can be induced by increasing the dislocation density. In addition to its effect on precipitation start and finish precipitation temperature, prior deformation also affects the volume fraction of precipitates; if Fig. 6 and Fig. 11 are compared, it is evident how grain and twin boundary precipitation of δ phase is more pronounced after 0.2 than after 0.005. Therefore, even though precipitation start and precipitation finish are relatively insensitive to deformation, the microstructural evaluation of the samples show significant differences between samples deformed in the elastic region and samples deformed in the plastic region.

Moreover, at high temperatures and deformed samples, there was also precipitation in the matrix, (as illustrated in Fig~~f~~. 11 and 12), which was not observed for elastically deformed samples. This difference between the characteristics of the precipitation with regard to the prior deformation was not detected in stress relaxation curves.

Below 875ºC two different stages of precipitation can be detected in the stress relaxation curves obtained for plastically deformed samples, whereas only one inflexion is detected for elastically deformed samples (see Fig. 7 and 13). In fact, according to the metallographic evaluation of the samples (Fig. 9 and 10), deformation would promote fast grain boundary precipitation, which would not be active for non-plastic deformation conditions. The fastest precipitation kinetics of these grain boundary precipitates would be around 875ºC and after six seconds. Under industrial conditions, this would mean that during the cooling of forged pieces, the regions with high dislocation densities, due to prior deformation would be prone to grain boundary precipitation. Another ~~likely~~ difference between elastically and plastically deformed samples at temperatures raging from 800ºC and 850ºC is the nature of the precipitates that form in the matrix. This precipitates would be γ'' for elastically deformed conditions (Fig. 4c) and some γ'

seems to appear for plastically deformed samples (Fig. 9c). However, this point should be better assessed by further metallographic evaluation.

When the relaxation curves of 0.1 and 0.2 deformation conditions are compared in Fig. 14, it is interesting to observe that the curves are very similar at high temperatures, where restoration mechanisms are taking place during relaxation. ~~In Fig. 15b it~~ It is evident ~~I~~in Fig. 15b that at 950ºC, both after 0.2 and 0.1 deformation, the steady state stress is reached and recovery is imminent in the first stages of relaxation. At low temperatures, recovery is more difficult, and therefore the relaxation rates are slower. Moreover, precipitation appears further reducing relaxation rates. The relaxation curves after 0.1 and 0.2 deformation separate; the relaxation curve after 0.2 deformation falls above the relaxation curve of 0.1 deformation. According to this, the lack of recovery at low temperatures implies that dislocation density is higher after 0.2 than after 0.1 deformation. Precipitation, induced by deformation, will increase its volume fraction the higher the dislocation density and, thus, relaxation rates will be diminished, which is the effect detected in the relaxation curves[j7].

5. Conclusions

A stress relaxation ~~based~~ technique has been used to characterize precipitation kinetics of Inconel 718 at temperatures within the hot deformation range. It has been shown that δ precipitation is relatively sluggish and it takes place at temperatures ranging from 850ºC and 950ºC. Prior ~~and~~ deformation increases the amount of δ phase which forms at grain and twin boundaries. At lower temperatures, precipitation at grain boundaries would only appear when prior deformation has been applied to the material and this precipitation is much faster. Grain boundary precipitation could have adverse effects on the hot ductility of the material. Even though Inconel 718 is usually deformed

at higher temperatures, special attention should be paid during forming processes to avoid long dwelling times at temperatures close 925ºC or slow cooling rates if the material has been previously deformed and has not been able to recover, i.e. reduce dislocation density.

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