# THE HIGH TEMPERATURE STABILITY OF IN718 DERIVATIVE ALLOYS

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## Abstract

The excellent fabricability and adequate heat resistant properties of IN718 have accounted for the widespread use of IN718 as a turbine engine disk material. The jet engine industry is placing new demands for an alloy, however, which exhibits the excellent fabricability and heat resistant properties of IN718, but which is not hampered by the alloy's relatively low temperature ceiling (approx. 650°C). **Ticolloy,** based on IN718, is an alloy developed through a systematic variation of the Al/Ti and (Al+Ti )/Nb atomic ratios. Mechanical property data has been gathered to document the tensile, crack growth, creep/stress rupture, and hydrogen susceptibility behavior of **Ticolloy** as compared to IN718. **Ticolloy** exhibited improved strength and high temperature stability, however, hydrogen susceptibility and crack growth behavior showed no improvement. Finally, a $\gamma$'' coarsening study has been carried out on a series of four new alloys. These alloys were developed to pursue the limit of the methodology employed to develop **Ticolloy**.

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## Introduction

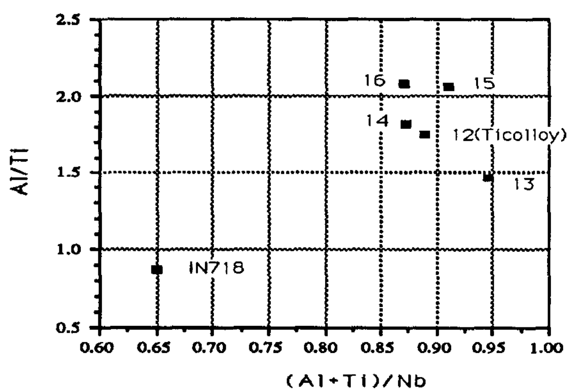
IN718 is a high strength, precipitation-hardened Ni-base superalloy that over a period of almost 30 years has gained huge acceptance for intermediate temperature applications in the aircraft industry. It accounts for roughly 35% of all actual wrought superalloy production. However, a set of goals outlined by the jet engine industry [1], which includes among its requirements the use of materials with higher temperature limit, higher hot corrosion resistance and lower density, is threatening the continued use of this alloy. One of the major limitations of IN718 is its lower ceiling temperature relative to other Ni-base alloys. It is known that this temperature limit is imposed by the microstructural instabilities that occur fairly rapidly at temperatures in excess of 650°C.

For several years, research [2-4] has been focused on elevating this ceiling temperature via the development of a more stable microstructure. The methodology used to develop **Ticolloy** is an increase of the Al/Ti and (Al+Ti)/Nb ratios in IN718 in order to achieve both a reduction in the coarsening rate of the metastable phase $\gamma''$ and a retardation on the formation of the more stable (equilibrium phase) $\delta$ phase [3].

The results presented herein are two-fold: first, preliminary baseline mechanical property data is presented to highlight the improved strength and microstructural stability of **Ticolloy.** Secondly, results of a coarsening study for four **Ticolloy** derivative alloys are presented in an effort to demonstrate a further improvement in microstructural stability over **Ticolloy.**

## Experimental

Figure 1 shows the chemistry of **Ticolloy**, standard IN718 and the series of four new alloys (13-16). As can be seen, not only were the Al/Ti and (Al+Ti)/Nb atomic ratios of the four new alloys increased with respect to **Ticolloy**, but the total strengthening elements (Al+Ti+Nb) also were increased. The rest of the composition (at.%)of all alloys is basically that of a standard IN718 alloy: Ni (52.52) + Fe(18.96) + Cr(19.91) + Mo(1.83) + C(0.15) + Al(1.91) + Ti(1.31) + Nb(3.40) is the average composition of Alloy 13. From this, Al, Ti and Nb contents were varied at the expense of Ni content.




| | Al+Ti+Nb (at%) |
|---|---|
| IN718 | 5.43 |
| Ticolloy | 6.75 |
| Alloy 13 | 6.62 |
| Alloy 14 | 6.87 |
| Alloy 15 | 7.23 |
| Alloy 16 | 7.47 |

Figure 1: Strengthening element atomic ratios of a standard IN718, Ticolloy and four new derivative alloys used in this study.

A master billet of Super IN718 was supplied courtesy of the Wyman-Gordon Company (WGC) to be used as the base material for IN718 and **Ticolloy** in initial material characterization. The billet was cut into smaller sections, vacuum induction melted (VIM) and alloyed to the desired **Ticolloy** chemistry courtesy of Special Metals Corporation (SMC). Alloys were also vacuum arc remelted (VAR) into ingots weighing approximately 15 pounds each. Forging was done courtesy of WGC. After initial characterization, an additional heat of **Ticolloy** produced at SMC and forged at WGC, and a heat of IN718, furnished by WGC, were used for tensile, creep/stress rupture, hydrogen susceptibility, crack growth, and heat treatment studies. The four new alloys used for the coarsening study each received VIM-VAR processing at Teledyne-Allvac. Subsequently these ingots were forged courtesy of the Cameron Forge Company.

Initial characterization involved hardness evaluation of samples aged up to 600 hours at 760°C. All other mechanical property tests were industry standard tests and specifications can be found elsewhere [5]. Tensile, creep and crack growth samples all received a commercial direct age heat treatment: 718°C 8 hrs/ FC 55°C per hour to 621/ 621°C 8 hours/ air cool. Samples for hydrogen susceptibility were heat treated using an industry heat treat schedule: 1037°C for 30 min AC/760 °C 10 hrs / FC 55 °C per hr / 648 °C 10 hrs/ AC.

Crack growth rates were measured for **Ticolloy** and IN718 at 650°C. Testing samples were industry standard $K_b$ bars notched on one surface. Crack length was then monitored in two directions. Tensile testing was done at several temperatures up to 788°C using non-ASTM standard specimens due to the limited availability of material. Creep/stress rupture tests were performed at 677 °C. To evaluate hydrogen susceptibility, both smooth and notched bar tensile samples were tested at ambient temperatures in both a 34.5 MPa hydrogen environment and a 34.5 MPa He environment.

For coarsening studies, small samples (1x1x2 $cm^3$) of alloys 13-16 were cut and then homogenized at 1120°C for one hour followed by water quenching. After homogenization the samples were aged at 760°C for times from 22 up to 600 hours to produce the strengthening phases γ' and γ''. The macroanalysis of the samples was done using optical microscopy (OM) as well as scanning electron microscopy (SEM). The microstructure of the heat treated samples was characterized using a transmission electron microscope (TEM) operated at 120 KeV and equipped with an energy dispersive spectroscopy (EDS) analyzer. The samples for the TEM study were prepared by a conventional jet polishing technique. In order to measure the true length of the γ'' particles the [001] zone axis was used to produce dark field images. About 700-2000 precipitates lying in two different $(100)_\gamma$ planes were used to calculate the average γ'' precipitate size. The calculation of the corrected sizes of precipitates was done following a conventional geometric technique [6].

## Results

### Mechanical Property Evaluation

Hardness data has been compiled for aged samples and results show that IN718 exhibits a reduction in hardness before 600 hours at 760°C whereas **Ticolloy** retains its hardness for at least that long, see Figure 2. Tensile data confirms a 25°C improvement in strength with comparable ductility to IN718 in the temperature range tested, see Figure 3.

Figure 4 shows the results of crack growth measurements. These results were inconclusive as to which alloy performed better. Crack rates measured in two directions, Figure 5, for both IN718 and **Ticolloy** show a possible orientation dependence. Crack growth in the "c" direction was slower for **Ticolloy**, however, crack growth in the "a" direction was faster.

Because **Ticolloy** has a higher Al content than IN718, the direct aged IN718 heat treatment does not adequately precipitate out all γ' phase. Hence, an optimized heat treatment for **Ticolloy** was determined. Hardness results confirm that employing an aging schedule of 760°C 8 hours / FC 55°C per hour to 648°C / 648°C 8 hours / air cool yields a **Ticolloy** with the highest hardness without δ formation, Figure 6. This modified heat treatment may further improve some of the mechanical properties of **Ticolloy**.

Hydrogen susceptibility was evaluated for **Ticolloy** using an industry heat treatment that approached the optimized **Ticolloy** heat treatment. As the results in Table I indicate, **Ticolloy** had comparative absolute tensile strength, and greater ductility than IN718. However, normalized data showed a poorer performance for **Ticolloy** than for IN718. Hence, **Ticolloy** has greater $H_2$ susceptibility than IN718.

The comparative results for Ticolloy and IN718 stress rupture lives show a slight improvement over IN718 in rupture times at 677°C, see Figure 7.

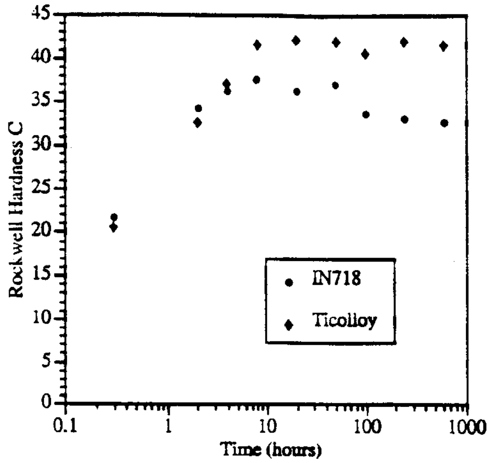



Figure 2: Rockwell C hardness data for IN718 and Ticolloy aged at 760 °C for different times. A plateau in the hardness for Ticolloy is observed where there is a decrease in hardness for IN718.

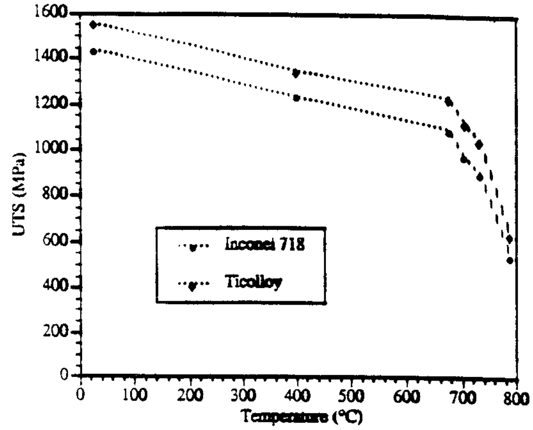



Figure 3: Ultimate Tensile Strengths for IN718 and Ticolloy for temperatures from ambient to 788 °C.

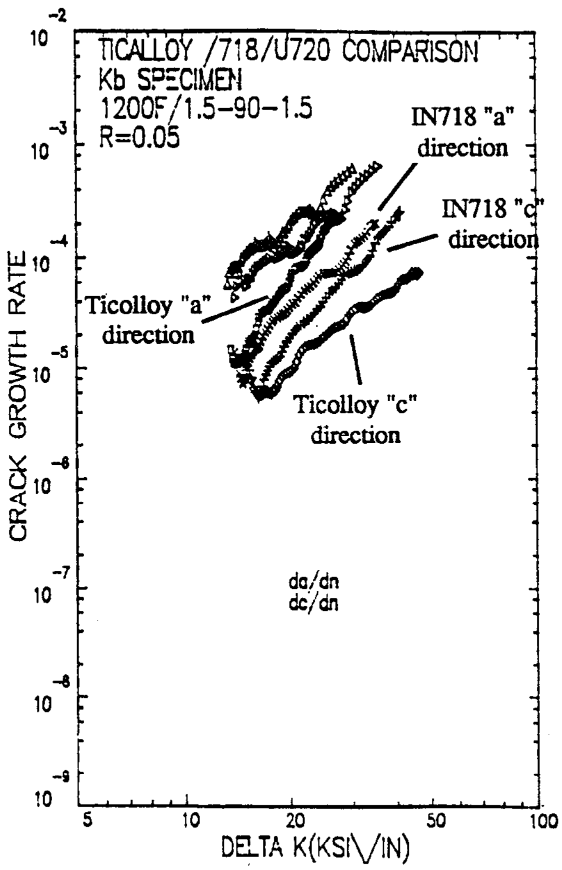



Figure 4: Crack growth data for IN718 and Ticolloy run at 650 °C, R=0.05, with 1.5 sec to load, 90 sec hold on load, and 1.5 sec to off load.

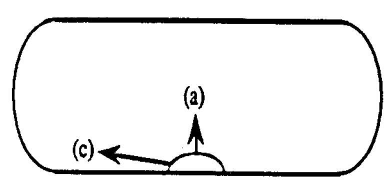



Figure 5: Cross section of crack growth specimen showing "a" and "c" directions of crack growth.

Table I:

**Tensile data from hydrogen susceptibility tests conducted at ambient temperature, and 34.5 MPa $H_2$ and 34.5 MPa He environments.**

| Specimen | Environ. | Ultimate Strength (MPa) | Reduction of Area (%) |
|---|---|---|---|
| Unnotched: | | | |
| Ticolloy | $H_2$ | 1386.4 | 14.3 |
| IN718 | $H_2$ | | 1.1 |
| Ticolloy | He | 1449.4 | 41.5 |
| IN718 | He | | 3.0 |
| Notched: | | | |
| Ticolloy | $H_2$ | 1223.2 | |
| IN718 | $H_2$ | 1590.0 | |
| Ticolloy | He | 1889.4 | |
| IN718 | He | 2220.0 | |

| | Notched Bar UTS $H_2$/He Ratio | Smooth Bar RA $H_2$/He Ratio |
|---|---|---|
| Ticolloy | .64 | .34 |
| IN718 | .72 [5] | .37 |

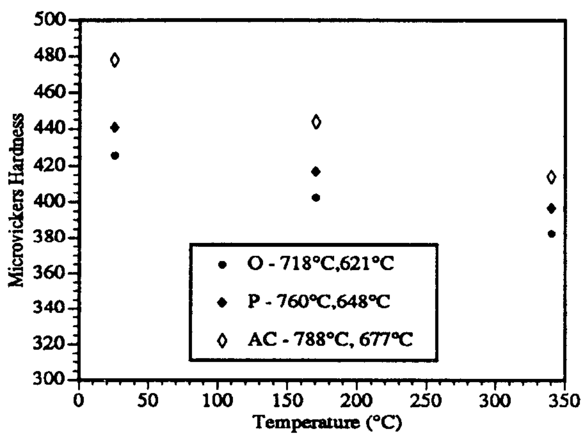



Figure 6: Hardness data for Ticolloy heat treated at various aging temperatures. Aging times were eight hours for each aging step.

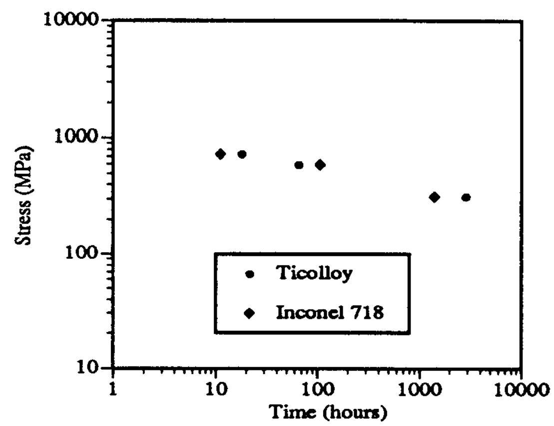



Figure 7: Stress rupture data showing improved lifetimes for Ticolloy. All tests were conducted at 677 °C.

## Coarsening Results

OM as well as SEM analysis revealed that the homogenization treatment used was sufficient to dissolve most second phase particles (all except carbides) and produce a homogeneous matrix made up of large (approx. 150 μm), equiaxed grains. It was noticed that a considerable number of twin boundaries were present in all samples after homogenization.

Selective area electron diffraction (SAED) and dark field imaging (DFI) also revealed that for ages up to 600 hours at 760°C the microstructures of the four alloys, 13 through 16 consist of spherical γ' ($L1_2$), disc shaped γ'' ($DO_{22}$) and plate-like δ ($DO_a$) particles dispersed in an fcc matrix.

As has been previously reported [3], the γ' particles consistently precipitated and grew on top of the γ'' particles. From a TEM analysis on samples with the 100 hours at 760°C aging treatment it was determined that the percent of γ'' particles which have a γ' particle attached to its surface increased from about 50% for the standard IN718 to almost 100% for the four new alloys. This trend holds for other aging times studied in this work. Figure 8 shows the microstructure of alloy 14 after 600 hours at 760°C. Figure 9 shows a high magnification image of the microstructure in same alloy. It is clear here that if there is a matrix layer between the γ' and γ'' particles it has to have a thickness of less than three unit cells (approx. 10Å).

Figure 10 shows the coarsening behavior of γ'' particles at 760 °C in IN718, **Ticolloy**, and in alloys 13 through 16. It is observed that particle coalescence had an effect in the average particle size in alloys 13 through 16 at long times. The data for IN718 and **Ticolloy** was obtained with coalesced particles treated as two or more individual smaller particles whenever it was possible to recognize them as such [3]. For the four alloys in the present study (13 though 16), coalesced particles were treated as single particles to take in account the effect of particle coalescence on the coarsening behavior of the γ'' precipitates.

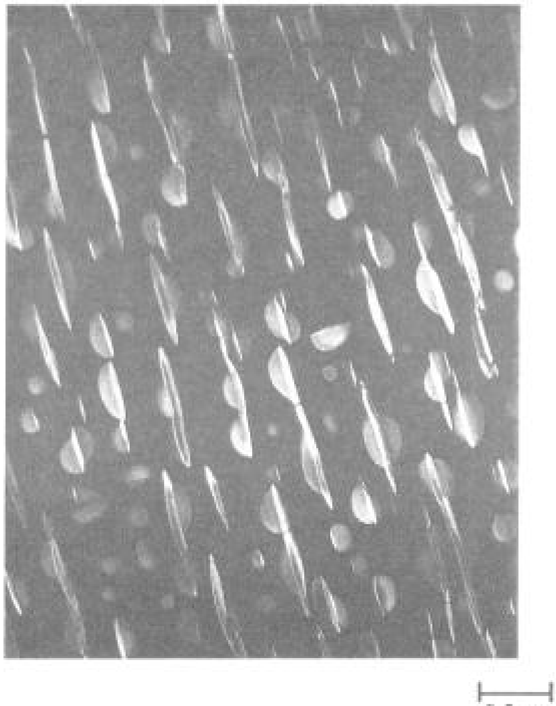



Figure 8: TEM micrograph showing the precipitate morphology in alloy 14 after 600 hours at 760 °C. Rounded particles are γ and disc-shaped particles are γ''. After 600 hours one of the three γ'' variants was dominant.

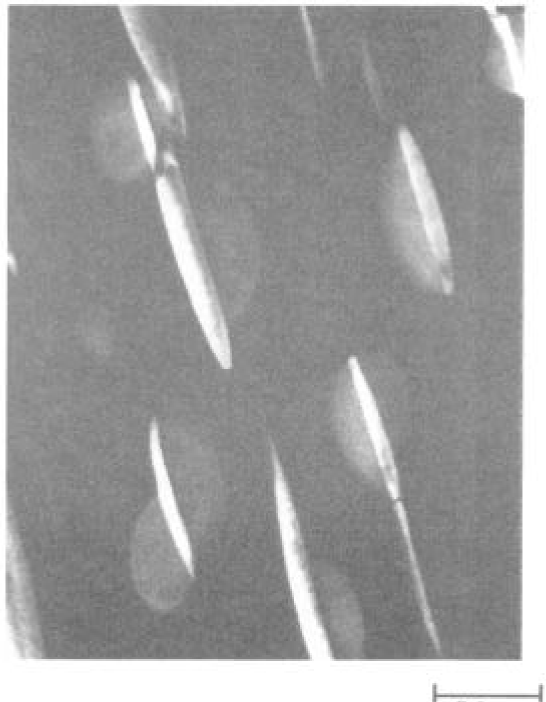



Figure 9: A high magnification TEM micrograph showing detail of γ/γ'' interface in same sample as Figure 8.

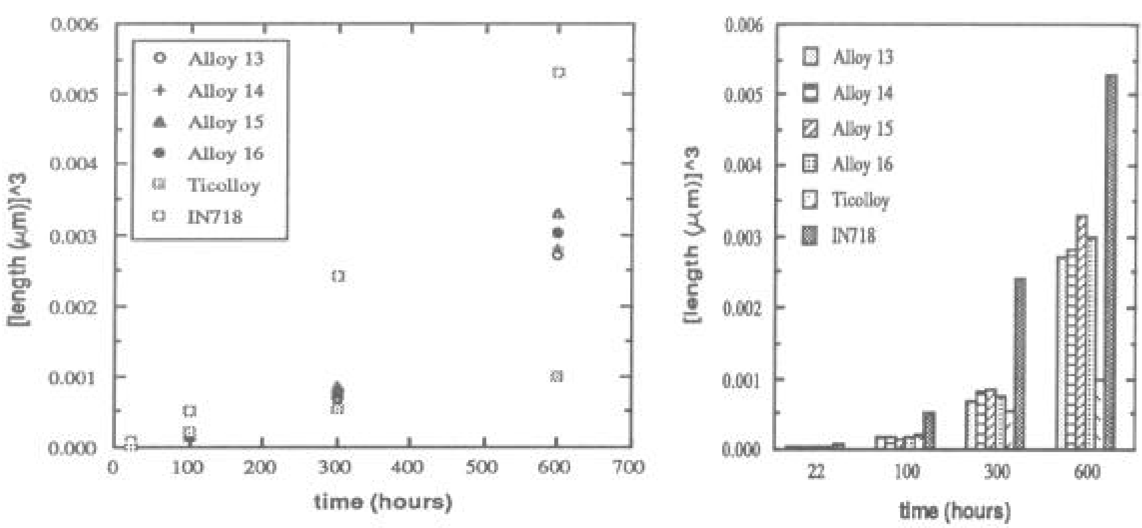



Figure 10: γ'' particle size at 760 °C as a function of time. Note that at 600 hours, the coarsening of γ'' in alloys 13 through 16 deviates from the linear behavior as predicted by the LSW theory. See text for the explanation of the high γ'' particle size in alloys 13 through 16 at 600 hours.

It is interesting to note that in alloys 13 through 16, one of the three different γ'' variants dominated and, as a consequence, grew faster while the other two variants were unstable and finally disappeared leaving grains with only one variant of γ'' each. Figure 11 shows this effect in alloy 14 aged for 22 hours at 760°C (three variants present) and in alloy 15 heat treated for 600 hours at 760 °C (only one variant present). For times longer than 22 hours the amount of δ was enough to be easily found in the TEM samples. Both grain boundary and intergranular δ precipitates were found in all four alloys. As it has been largely reported [7], zones denuded of γ'' were found around the δ particles. This is seen in Figure 12.

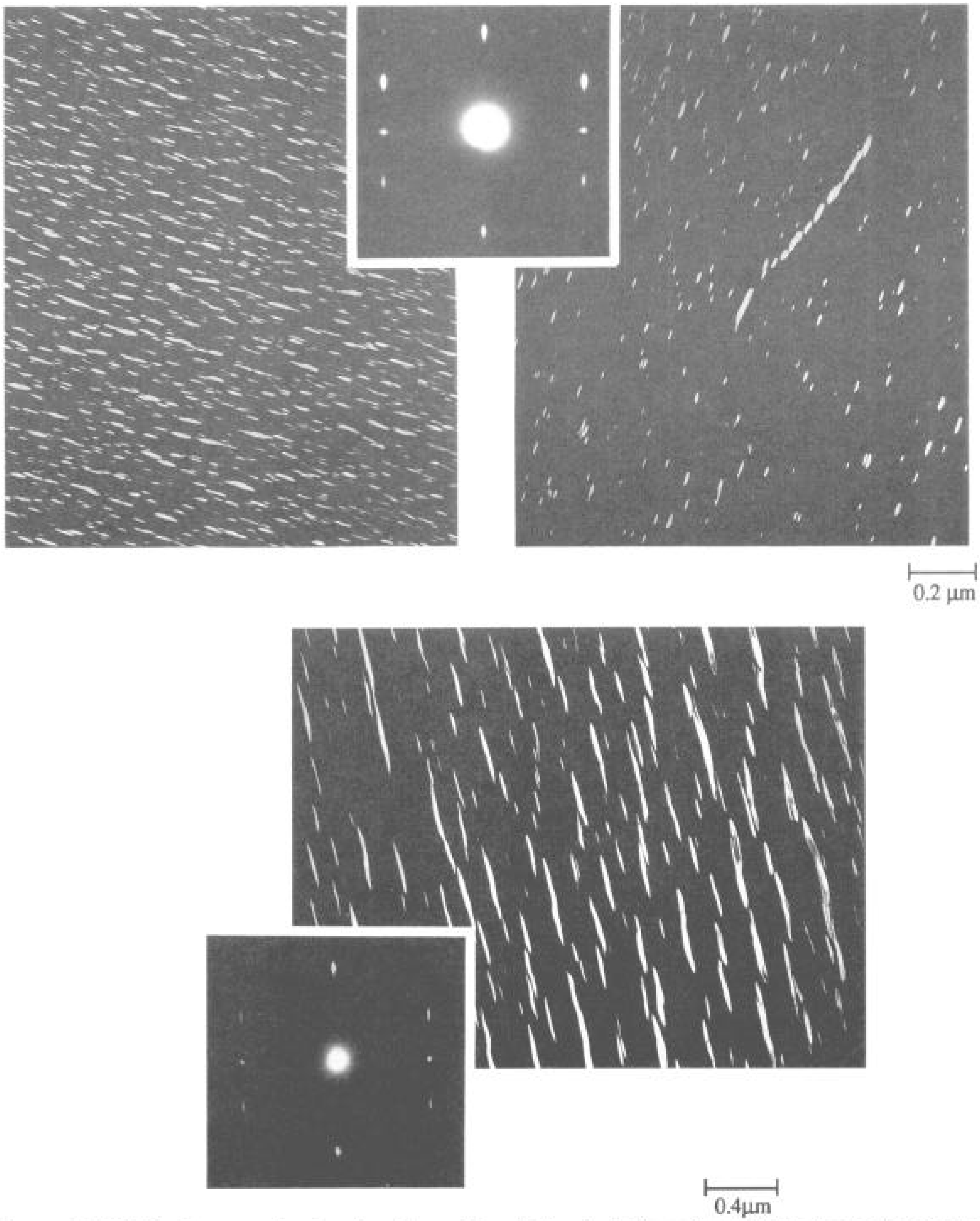



Figure 11: TEM micrographs showing the preferential orientation of one of the three γ'' variants (only two variants shown here) in sample 14 heat treated for 22 hours at 760 °C (top) and alloy 15 heat treated for 600 hours at 760 °C (bottom). The diffraction pattern is included to show the intensities of each variant.

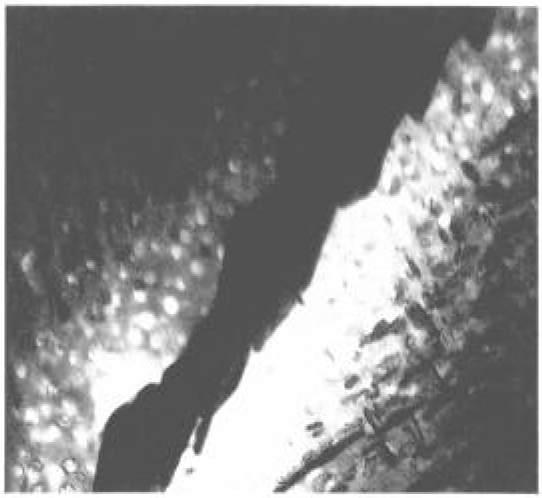



Figure 12: TEM micrograph showing the denuded zone around the grain boundary $\delta$ precipitate in sample 13 heat treated for 600 hours at 760 °C.

## Discussion

The plateau observed in the hardness data compiled for **Ticolloy** indicates that its microstructure is resisting better the instabilities intrinsic to the $\gamma''$ phase. This plateau can be compared to the decrease in hardness exhibited by IN718 within the same range of aging times and temperature. Similar behavior showing improved strength for **Ticolloy** was also confirmed with tensile data. Crack growth results are inconclusive due to the dependence of crack growth rate on direction. This might be attributed to the preferential precipitate growth direction which has become evident in alloys 13 through 16. The stress-rupture life behavior of IN718 has been well documented at several temperatures [8]. IN718 shows a break-off in expected rupture life which is attributed to $\gamma''$ dissolution and $\delta$ formation. In an effort to determine a similar dropoff for **Ticolloy**, stress rupture tests were run. The results showed a slight improvement in thermal stability due to reduction in coarsening kinetics for **Ticolloy**. However, more testing is required to sufficiently document this dropoff phenomenon. Hydrogen susceptibility data has shown that the presence of delta phase, which is greatly reduced in **Ticolloy**, may not be controlling the performance of the alloy in the hydrogen environment. Further testing is required to characterize the behavior.

The criterion on which the series of IN718 derivative alloys (including **Ticolloy** and alloys 13 through 16) were designed, is that by increasing the amount of $\gamma'$ phase precipitated on the surface of $\gamma''$ particles, the coarsening rates of those $\gamma''$ particles are effectively reduced. The reason for this is believed to be the fact that as the amount of $\gamma''/\gamma$ (particle/matrix) interface gets reduced, the diffusion of the Nb atoms from a small particle to a larger one as needed for $\gamma''$ coarsening becomes more difficult. This criterion proves correct if it is considered that in going from IN718 to **Ticolloy** there is a large increase in the percent of $\gamma'/\gamma''$ events, corresponding to a large reduction (approx. one order of magnitude) on the average $\gamma''$ coarsening rate [3]. Also, as in the new alloys 13 through 16 the increase in the percent of $\gamma'/\gamma''$ events gets much smaller as does the reduction percent of the $\gamma''$ coarsening rates.

The increase in the $\gamma''$ particle sizes on alloys 13 through 16 for times longer than 300 hours is believed to be caused by the coalescence of particles. As seen in Figure 11 , for long times one $\gamma''$ variant becomes dominant and as a consequence the combined effect of assisted growth and coalescence results in a larger particle size. Because of this high particle coalescence, the coarsening of the γ'' particles at long times does not follow the linear behavior predicted by the Lifshitz-Slyozov-Wagner (LSW) theory [9,10]. Thus, coarsening rates calculated including data from long time agings would result in values larger than the actual values due only to particle coarsening. The effect of coalescence on the coarsening behavior of γ'' particles in IN718 alloys has been already reported [11]. The reason for the γ'' variant preference observed in these four new derivative alloys is not clear at this point, however recent results to be published suggest that this phenomenon might be caused by elastic effects.

IN718 was designed as a γ'' strengthened superalloy and therein lies the reason for its limited operating ceiling temperature. The preliminary results on mechanical behavior of **Ticolloy** support the hypothesis that the mechanical properties of IN718 can be improved by promoting a more stable microstructure. Preliminary results on the γ' coarsening study in the four new derivative alloys indicate that the coalescence of particles might be an important factor to consider in the search for thermally stable microstructures with a high content of second phase forming elements. Further mechanical property characterization of alloys 13-16 will determine whether these IN718 derivatives can ultimately be considered as replacement alloys for IN718.

## Summary

1.- Using the same commercial direct aging heat treatment, **Ticolloy** exhibited improved thermal stability than IN718 and this translated into some improved mechanical properties. A 25°C improvement in strength with comparable ductility to IN718 was obtained. The improvement on crack growth is ambiguous due to a preferential direction for crack growth. For hydrogen susceptibility tests, absolute data shows comparable strength and improved ductility, however, normalized data shows no improvement for **Ticolloy**.

2.- An optimized direct-aging treatment was developed for **Ticolloy**. Hardness data show that this new heat treatment may give **Ticolloy** still better mechanical properties.

3.- The γ'' coarsening behavior on four new IN718 derivative alloys was evaluated. These alloys have higher Al/Ti and (Al+Ti)/Nb atomic ratios as well as higher total (Al+Ti+Nb). For times longer than 22 hours, alloys 13 through 16 showed a microstructure with a preferred γ'' variant. At about 600 hours all four alloys showed grains with only one of the three different γ'' variants present. The γ'' particles sizes were at least comparable to those in **Ticolloy** for times up to 300 hours. After 300 hours the coalescence of γ'' particles caused a rapid increase in the average size.

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