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Effects of Tin on Microstructure and Mechanical Behavior of Inconel 718

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INTRODUCTION

Tin (Sn) has generally been viewed as potentially deleterious to nickel-base superalloys. It has been classified with other tramp and trace elements (refs. 1 and 2), although a limited investigation of Sn additions failed to confirm the deleterious effect of Sn in a wrought nickel-base superalloy (refs. 2 and 3). Tin (group IVB) is so classed because of its chemical similarities to lead (group IVB) and bismuth (group VB), both of which have been shown to be deleterious to nickel-base superalloys (refs. 1 to 3) even at low parts per million concentrations. This investigation has examined the effects of Sn additions on the superalloy with the greatest demand (ref. 4) for columbium (Cb), Inconel 718.

Incomel 718 contains about 5-1/2 percent Cb, which may be associated with Sn in its native state. Lower grades of commercial ferrocolumbium may contain as much as 0.25 percent Sn (ref. 5). This investigation has evaluated the effects of Sn on Incomel 718 at levels that might be typical of and that greatly exceed those anticipated if a lower purity grade of ferrocolumbium is used as melting stock.

EXPERIMENTAL PROCEDURE

Material Preparation

The starting material, except as noted later, was a production billet of Inconel 718. The billet was cut and remelted in a 14 kg vacuum induction furnace, the desired Sn additions were made as late additions. The composition of Inconel 718 is shown in table I. The intended Sn levels and the actual Sn level determined by two chemical analysis methods are shown in table II. The desired levels of Sn addition from 50 to 10 000 ppm were accurately retained.

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Two 7-cm-diameter ingots were poured from each heat. The ingots were rolled as follows:

Initial temperature, 1090±15 °C Reductions per pass

Initial heat 0.38 cm in 3 passes Reheat 0.38 cm in 3 passes Reheat 0.38 cm in 3 passes

Heat treatment at 980 °C for 30 min was followed by reduction to final size and an air cool to room temperature. Final rolldown dimensions were approximately 6.4 cm wide by 30 cm long by 1.3 cm thick.

Test specimen blanks were cut from the rolldowns and subjected to the following heat treatment:

950 °C for 1 hr and air cool to room temperature

720 °C for 8 hr and cool at 50 °C/hr to

620 °C for 8 hr and air cool to room temperature

Mechanical Testing

Tensile tests were performed at room temperature, 425, and 650 °C using 63-mm-diameter cyclindrical specimens. The tests were performed in accordance with ASTM recommended practices E8 and E21. Duplicate tests were performed at 650 °C on material in the as-heat-treated condition. The 650 °C tests were also performed on heat treated material which was given a 250-hr soak at 650 °C. At room temperature both longitudinal and transverse tests were performed. Data reported for 425 °C tests is for individual tests unless otherwise noted. These bars were erroneously heat treated initially then reheat treated according to the above schedule in flowing argon. They were tested with a thin oxide scale resulting from the heat treatment.

Constant-load stress-rupture tests were performed at 650 °C and stresses of 469, 579, and 793 MPa in accordance with ASTM Recommended Practices E139 and E292. The tests at 793 MPa were performed on combination smooth and notched bars, the notch having a K_{t} of approximately 3, and were performed on both heat treated material and heat treated material which had been suaked for 250 hr at 650 °C. Other tests were performed on bars of a smooth cylindrical test section.

RESULTS AND DISCUSSION

Tensile Tests

Tensile tests were performed at room temperature, 425 and 650 °C. The results of room temperature tests are summarized in figure 1, which shows that the average ultimate strength is virtually constant at 1420 MPa and that the yield strength is constant at 1196 MPa as the Sn concentration is varied to as much as 1 wt %. It should be noted that the ultimate strength value of

1402 MPa for the 800-ppm level falls slightly below the lower 99 percent confidence limit of 1409 MPa for the data. The room-temperature ductility summarized in figure 1 shows that the lowest levels of both elongation and reduction in area are at 1 wt %. The 20.5 percent elongation and 42.6 percent reduction in area are below the 99 percent confidence levels of 22.8 and 45.6 percent, respectively. We suggest that these slightly reduced ductility levels might be caused by the microfissuring of the 1 wt % Sn alloy (fig. 2).

Not shown are the results of transverse tensile tests performed at room temperature. The ultimate and yield strengths were independent of Sn content and averaged 1400 and 1157 MPa, respectively. The transverse room-temperature ductility averaged 20 percent elongation and 32 percent reduction in area. The lowest values were for the virgin heat with no Sn added. No correlation with Sn levels could be noted.

Also not shown are the results of 650 °C tensile tests of samples aged 250 hr at 650 °C. For alloys of 800 ppm Sn, or less, the greatest property degradation observed was a 5 percent relative decrease in elongation. No correlation with Sn content and property change after aging was noted.

The results of tensile tests performed at 425 °C are summarized in figure 3. The average ultimate tensile strength is 1276 MPa, and the average yield strength is 1119 MPa. At 1 wt % Sn, one test (ultimate tensile strength, 1204 MPa; yield strength, 1079 MPa) is statistically below the average (99 percent confidence) for both the ultimate and yield strengths. Also one yield strength value at 420 ppm Sn (1079 MPa) was statistically significantly below the average. The reduction in area values of both tests and one elongation value (17 percent) at 1 wt % Sn were statistically significantly below the average values of 19.4 and 40.3 percent, respectively. The 17-percent elongation measured for one test with 210 ppm Sn was also significantly below the average value.

Figure 4 summarizes the strengths measured at 650 °C. The ultimate strength appears to be independent of Sn level and has an average value of 1127 MPa. The lowest individual measurement was 1104 MPa, obtained for a test bar with 90 ppm Sn. Similarly, the yield strength appeared to be independent of Sn amount, being nearly constant at 972 MPa and with the minimum individual value of 930 MPa occurring for a sample with 800 ppm Sn. Ductility appeared to vary with Sn levels having greater values between 70 and 420 ppm than at either the very low or 1 wt % Sn levels. The elongation measurements of 13.4 and 14.6 and reduction of area of 18.2 and 14.3 for 1 wt % Sn samples are below the 99.9 percent confidence level for the mean of the data. As mentioned previously, this is attributed to the microfissures observed in 1 percent Sn samples.

Stress Rupture Tests

Stress-rupture tests were performed on combination smooth and notched bars at 650 °C with an applied stress of 793 MPa. Additional tests were performed on smooth bars at 469 and 579 MPa. The average of the duplicate tests at 793 MPa shown in figure 5(a) shows no adverse effect of Sn at or below 800 ppm. The maximum scatter of these data for duplicate tests was 10.5 hr. At 1-wt % Sn one bar had a life of only 2.3 hr; the other 34.3 hr. However, both failed in the notched section. None of the lower Sn level bars broke in

the notched section. All test bars had rupture lives equal to or greater than 33.6 hr. Barker, et al. (ref. 6), reported a life of 10.6 hr for Inconel 718 tested at the same test condition. The results of all the rupture tests are compared with those from reference 6 in figure 5(b). The results for Inconel 718 containing 800 ppm or less Sn, compare well with those of reference 6. For example, at 468 MPa, the range of the data for the tests in this investigation is 5752 to 6189 hr. Reference 6 reported a single data point of 7263 hr. In terms of Larson-Miller parameter, at that test condition the difference is 0.17 parameter, for the shortest life test of our set compared with reference 6. At the higher stress tests (579 MPa) the lives measured in this investigation appear to be greater than would have been expected based on the results in reference 6. The rupture ductility for Inconel 718 with 800 ppm Sn or less appears to be independent of the Sn content. As can be seen in figure 5(c), the 1-wt % Sn alloy had lower ductility than the lower Sn level materials.

Combination smooth and notched bars soaked for 250 hr at 650 °C were also stress-rupture tested at 650 °C with an applied stress of 793 MPa. The rupture lives were greater than for the unaged material. Ine ductilities generally were also greater than for the as-heat treated condition; however, the 1 wt % Sn alloy still fractured in the notch. No other property changes were related to Sn content.

Metallurgical Evaluations

As heat treated samples were examined by light microscopy. Typical photomicrographs are shown in figure 6. No significant differences can be seen among the microstructures of the alloys of differing Sn contents. After approximately 6000 hr of exposure to 650 °C, no significant difference in microstructures were observed (fig. 7).

Examination was also performed using transmission electron microscopy, scanning electron microscopy, and x-ray diffraction analysis of extracted residues. No differences in structure were observed among samples of differing Sn levels nor were any phases observed that have not previously been reported for Inconel 718 having similar thermomechanical history. For the 1-wt % Sn alloy the plate-like orthrhomoric Ni₃Cb appeared to have a slightly higher Sn content than the matrix. A typical analysis of the residue electrolytically extracted with 1 wt % ammonium sulfate plus 1 wt % citric acid in water from material exposed about 6000 hr at 650 °C is Ni, 70; Cb, 16-1/4; Ti, 5-1/4; Al, 3-1/2; Cr, 2, Fe, 2, and Mo, 1 at %. The samples yielded about 20 wt % of such residues. No significant differences in Sn levels were observed in these extracted residues.

CONCLUDING REMARKS

This investigation was performed to evaluate the effects of Sn on the behavior of Inconel 718 with a view toward considering the use of a lower grade of ferrocolumbium as melting stock if vacuum grade Cb availability should become scarce. The results of this investigation suggest that in amounts less than about 800 ppm, Sn does not greatly degrade Inconel 718. However, occasional statistically significant low values of strength or ductility were noted at Sn contents as low as 200 ppm for a remelt heat and 90 ppm for a virgin heat.

The reason for these low values was not established by this investigation. The results suggest that Inconel 718 is reasonably tolerant of Sn additions and that further investigation is warranted. Particular attention to the effects of larger billet size on Sn segregation is suggested to assure that the high Sn regions do not become prone to microfissuring as was seen at the 1-wt % Sn level in this work. Care should also be taken relative to the amount of lead which night be introduced, particularly if low-alloy steel grade ferrocolumbium (ref. 5) is considered for use, as it may contain up to 0.25 percent lead, which is known to be highly detrimental to Ni-base superalloys. Also low and high cycle fatigue evaluation should be conducted before application in critical components.

The results of the investigation are summarized as follows:

- 1. The effect of Sn on tensile properties at room temperature 425 and 650 °C is small for Sn levels of 800 ppm or lower. For 1 wt % Sn both elongation and reduction in area were lower than for the lower Sn level alloys.
- 2. Stress rupture lives at 650 °C at applied stresses of 469, 579, and 793 MPa were unaffected by 800 ppm or less Sn. At 1 wt % Sn the life of one of two tests bars at 793 MPa was greatly reduced, and both bars failed in the notch section of combination notched and smooth test bars. At 469 MPa the ductility of 1 wt % Sn test bars was lower than that of lower Sn levels.
- 3. Microfissuring was observed in Inconel 718 containing 1 wt % Sn. We believe the lower ductility generally observed for the 1 wt % Sn alloy is caused by the microfissuring.
- 4. Metallographic and x-ray diffraction analyses of as-heat treated and tested material revealed no new phases or unusual microstructures.

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TABLE I. - INCONEL 718 COMPOSITION

| Elemena | Content, wt % | Element | Content, wt % |
|---------|------------------|---------|-------------------|
| Fe | Balance | С | c _{0.10} |
| Cr | 17.00 - 21.00 | Mn | C.35 |
| Nb+Taa | 5.00 - 5.50 | Si | C.35 |
| Mo | 2.80 - 3.30 | Р | c.015 |
| T1 | .65 - 1.15 | S | c.15 |
| Al | .40 - 0.80 | B | c.006 |
| N1+Cob | 50.00 - 55.00 | | |

^aTa is typically less than 0.1 percent. bCo is typically less than 1 percent. CMaximum.

TABLE II. - ALLOYS INVESTIGATED

| Nominal Sn addition to | Sn content, ppm, determined by | | |
|---------------------------|--------------------------------|-------------------|--|
| Incomel 718, | Photoemission spectrograph | Atomic absorption | |
| (a) | <5 | <5 | |
| (a) | 90 | 88 | |
| b+0 | 10 | 50 | |
| b ₊₅₀ | 70 | 83 | |
| b ₊₁₀₀ | 120 | 88 | |
| p+500 | 210 | 188 | |
| b ₊₄₀₀ | 420 | 375 | |
| b+800 | 800 | 750 | |
| +1.0 wt % | 9900 | 7900 | |

aVirgin heats of different ferrocolumbium grades.

bCommercial alloy heat with Sn additions to remelt.

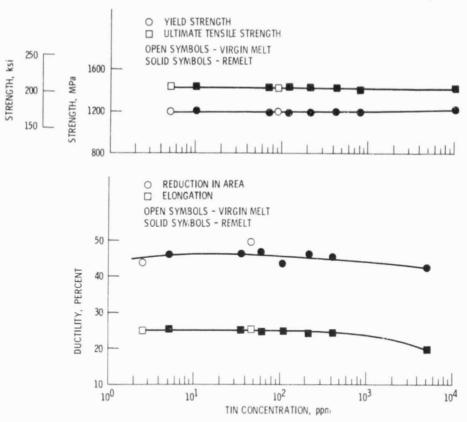


Figure 1. - Effect of tin on longitudinal room-temperature tensile properties of Inconel 718.

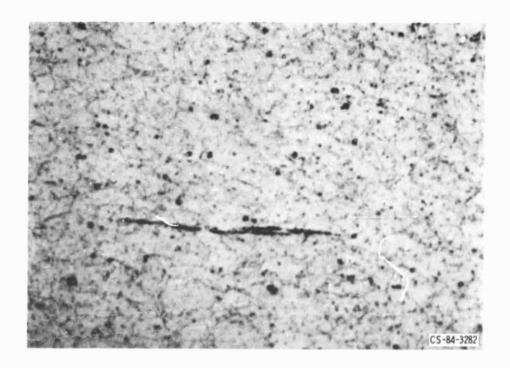


Figure 2. - Microfissuring in Inconel 718 with 1 wt % Sn.

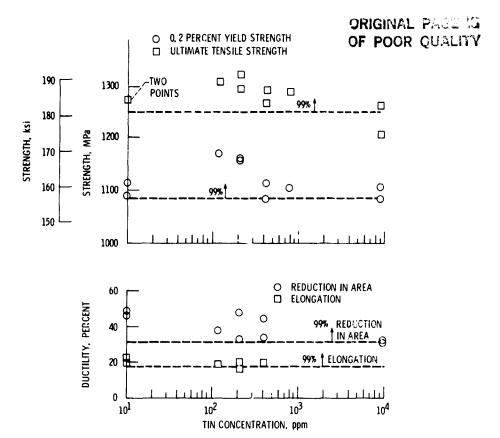


Figure 3. - Effect of tin on 425 ^OC longitudinal tensile properties of Inconel 718.

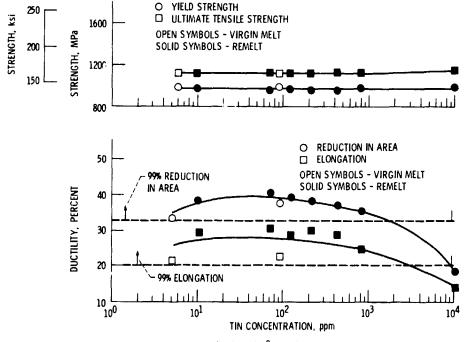


Figure 4. - Effect of tin on longitudinal 650 $^{
m O}{
m C}$ tensile properties of Inconel 718.

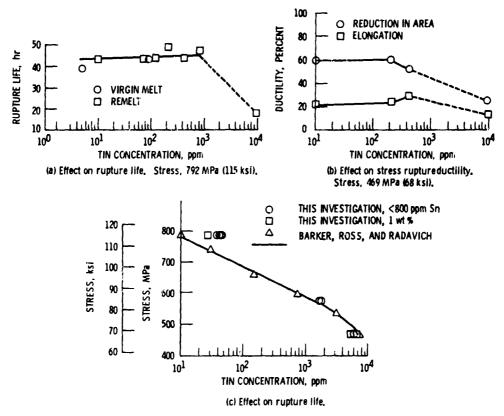
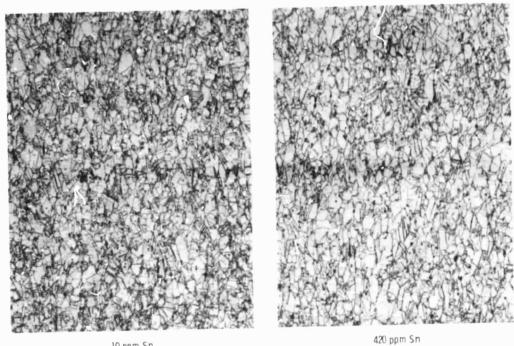


Figure 5. - Effect of tin content on 650 °C stress rupture properties of Inconel 718.

ORIGINAL PACE 19 OF POOR QUALITY



10 ppm Sn

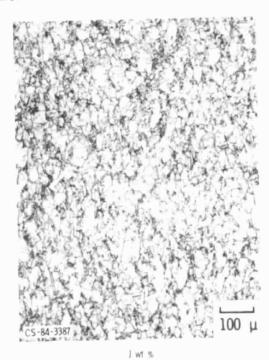


Figure 6. - As-heat-treated Inconel 718.

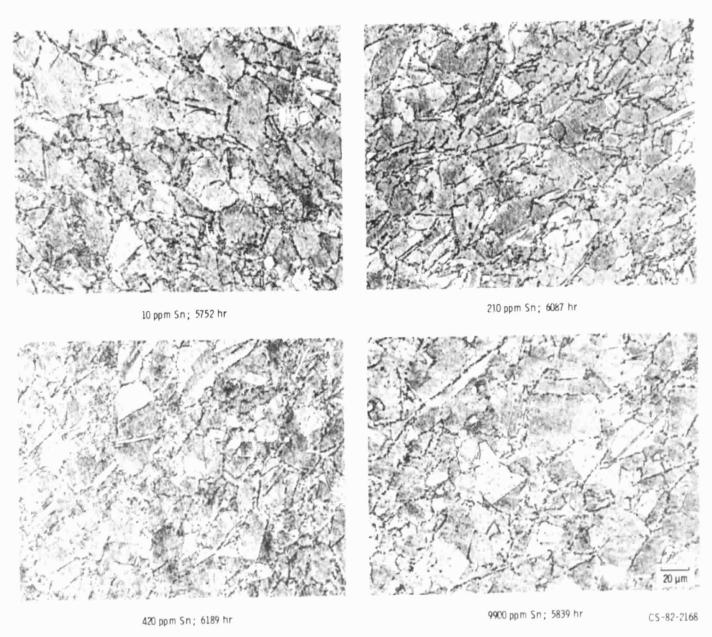


Figure 7. - Incomel 718 after stress-rupture test at 650 °C. Etchant: 35 ml ethanol, 65 ml hydrochioric acid, and 7 drops hydrogen peroxide.