

Assessment on the Thermo-mechanical Fatigue Properties of 98 Ni-base Single Crystal Superalloys

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Abstract

Thermo-mechanical fatigue (TMF) property data generated in NIMS on 1st to 5th generation single crystal (SC) superalloys have been analyzed to understand the compositional and structural factors affecting the property. It was found that Re and Ru additions effectively improve the out-of-phase TMF property. The optimum γ' fraction was found to be about 74%. A prediction equation for the TMF life from a given chemical composition has been derived for designing further superior SC superalloys.

Introduction

Thermo-mechanical Fatigue (TMF) of single crystal (SC) turbine blade materials is becoming a critical issue, because of the ever increasing inlet gas temperature and the stronger internal cooling, causing a higher thermal stress in the airfoil material [1-3]. Moreover, when used in land-base gas turbines in weekly or daily start and stop (WSS or DSS) mode - the latter is most typical in Japanese gas-turbine power stations - the turbine airfoil materials suffer from a larger number of TMF cycles. In such situation, SC superalloys with higher TMF properties rather than creep properties are desired by turbine designers.

Since our early work published in Superalloys 2004 [1], we accumulated TMF data of so far 98 commercial or experimental SC superalloys, including 1st to 5th generation alloys. This paper reports the analysis of the effect of compositional and structural factors on the TMF property. The guideline for designing TMF resistant SC alloys will also be discussed.

2. Experimental Procedure

2.1 Single crystal alloy specimens

In total 98 different SC superalloys, from 1st to 5th generation alloys, have been tested. The alloys include commercial/practical alloys such as ReneN4, ReneN5, CMSX-4, CMSX-10, PWA1480, PWA1484, MX-4/PWA1497, and also NIMS alloys such as TMS-6, TMS-75, TMS-82+, TMS-138, TMS-138A, TMS-196 and so on.

The chemical composition range of SC superalloys examined in this study are presented in **Table 1**, with the typical NIMS SC superalloy compositions. All the alloys were cast in NIMS in house directional solidification furnace, solution and aging heat treated also in NIMS vacuum furnace. Specimens with their longitudinal directions within 5° of $\langle 001 \rangle$ orientation were machined for the TMF test. The gauge part of the specimens was 5 mm in diameter and 15 mm in length.

2.2 TMF testing condition

TMF testing condition was the out-of-phase condition simulating airfoil materials in land-base gas turbines. Servo-hydraulic, closed-loop machines, MTS-type 810, using radio frequency induction heating system in air have been used. **Fig.1** shows a schematic plot of the out-of-phase TMF cycling, in which, ϵ_t is total strain, T is temperature, hold time is 1 hour at 900°C in the compression stage. The total cycle period is 66 min. The total strain was measured using an axial extensometer on the gauge length to 12 mm.

Calibrations were performed on the thermal expansion of specimens with temperatures. A zero strain was applied at the mid-temperature of 650 °C before starting the TMF cycling.

A simple tensile test at 400°C was also performed with almost all the alloys to help analyzing the TMF property.

Table 1 Chemical composition range (at%, bal. Ni) of SC superalloys examined in this study, with typical NIMS alloy compositions.

| | Co | Cr | Mo | W | Al | Ti | Ta | Hf | Re | Ru |
|-----------------------|--------|--------|-------|-------|-----------|--------|-------|--------|-------|-------|
| SC Alloy | 0~18.0 | 0~12.3 | 0~3.8 | 0~5.3 | 6.90~18.7 | 0~5.06 | 0~3.8 | 0~0.16 | 0~6.3 | 0~3.8 |
| TMS-6 (1st gene) | 0 | 10.9 | 0 | 2.9 | 12.2 | 0 | 3.5 | 0 | 0 | 0 |
| TMS-82+ (2nd gene) | 8.2 | 5.8 | 1.2 | 2.9 | 12.2 | 0.63 | 2.1 | 0.04 | 0.8 | 0 |
| TMS-75 (3rd gene) | 12.5 | 3.5 | 1.2 | 2.0 | 13.7 | 0 | 2.0 | 0.04 | 1.6 | 0 |
| TMS-138 (4th gene) | 6.1 | 3.8 | 1.8 | 2.0 | 13.6 | 0 | 1.9 | 0.04 | 1.6 | 1.2 |
| TMS-196 (5th gene) | 6.0 | 5.6 | 1.5 | 1.7 | 13.1 | 0 | 1.9 | 0.04 | 2.1 | 3.1 |

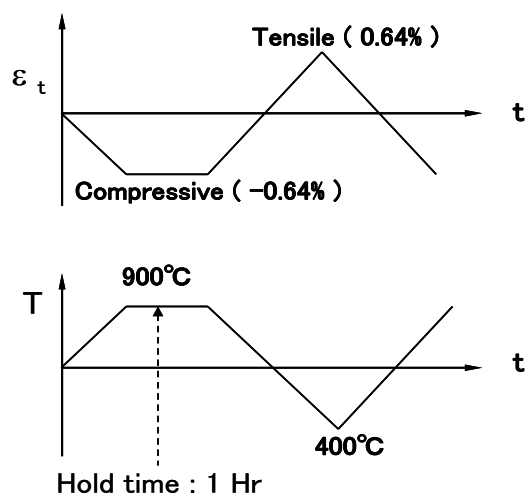


Fig.1 Schematic diagram of out-of-phase TMF cycle.

3. Results and Discussion

3.1 Effect of alloy composition

Typical 1st cycle hysteresis loops of (a)TMS-144 ruptured in the 21st cycle and (b)TMS-196 ruptured in the 198th cycle are presented in Fig.2. The large difference in cycles to failure comes firstly from the difference in stress relaxation during the hold time under compression at 900°C. The large relaxation under compression with TMS-144 causes a yielding under following tension condition at 400°C, resulting in a large hysteresis loop and giving a serious damage in the microstructure every cycle. On the other hand, TMS-196 containing 3.1at%Ru and 2.1at%Re is very resistant to the compression relaxation and only a small amount of yielding

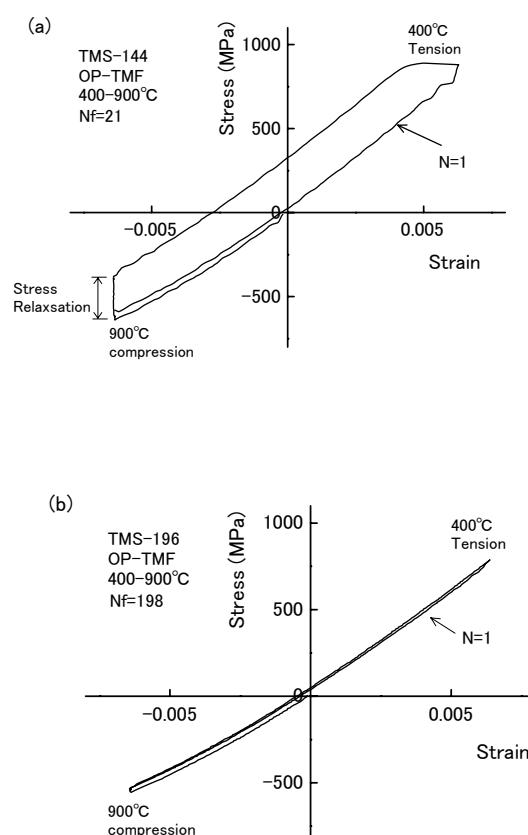


Fig. 2 Example of typical hysteresis loops at first cycle for Ni-base SC superalloys, (a) fractured in 21st cycle and (b) in 198th cycle.

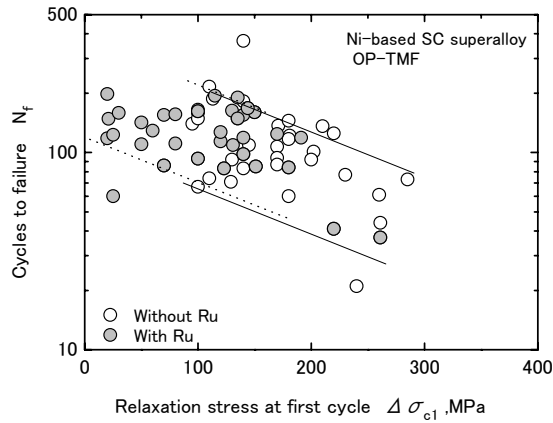


Fig.3 Relation between relaxation stress at first cycle and cycles to failure.

occurred in the first cycle. In the later cycles the stress relaxation became larger but still much smaller than the case of TMS-144. **Fig.3** shows a relationship between stress relaxation at first cycle and cycles to failure, showing a general tendency that alloys with smaller stress relaxation provide better TMF lives. The stress relaxation is thus clearly found to be one of the most important factors to govern the out-of-phase TMF property as suggested in our previous paper [1].

Fig.4 shows that alloys with higher Ru contents in general are more resistant against the relaxation at 900°C. The stress relaxation can be minimized when Ru is added up to 4 at%. **Fig.5** shows that Re, as well as Ru, is a very effective strengthener against the relaxation. Both Ru and Re metals are partitioned more to γ phase rather than γ' phase. Also both of them have the hcp structure, and naturally decrease the stacking fault energy in γ phase when added to superalloys. As shown in **Fig.6**, Zhou and some of the present authors [4] found that the lower stacking fault energy stimulates dislocation dissociation to form stacking faults even in the thin γ matrix channels in a 4th generation alloy TMS-138, which reduced the dislocation mobility and improved LCF property. The same mechanism seems to be operating during the relaxation process, resulting in the excellent TMF property in the 4th and 5th generation SC alloys containing Ru and Re.

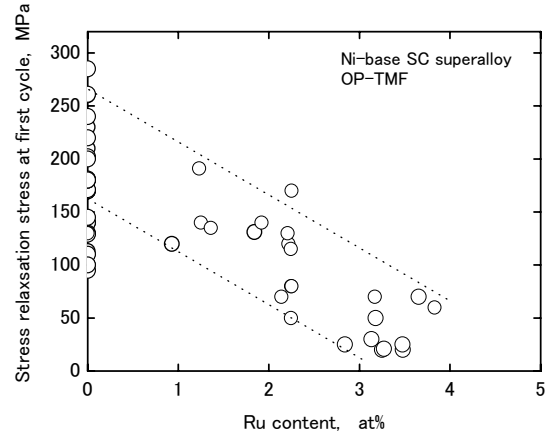


Fig.4 Relationship between Ru content and the stress relaxation at first cycle, showing new generation SC superalloys with Ru additions have advantages in the TMF property.

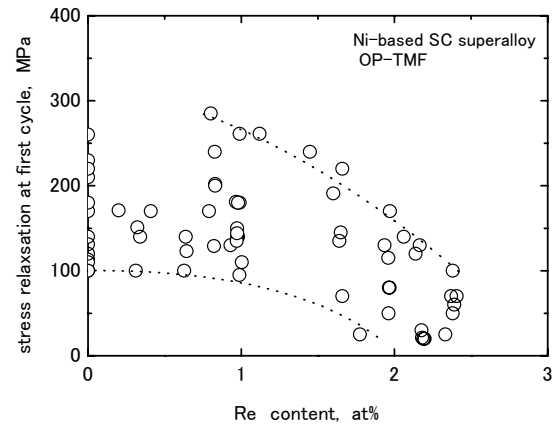


Fig.5 Relationship between Re content and the stress relaxation at first cycle, showing new generation SC superalloys with Re additions have advantages in the TMF property.

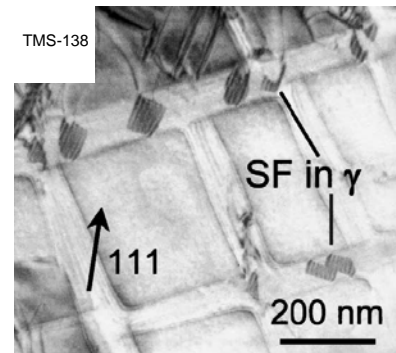


Fig.6 TEM photograph of dislocation and stacking faults taken from a fatigued specimen of TMS-138.

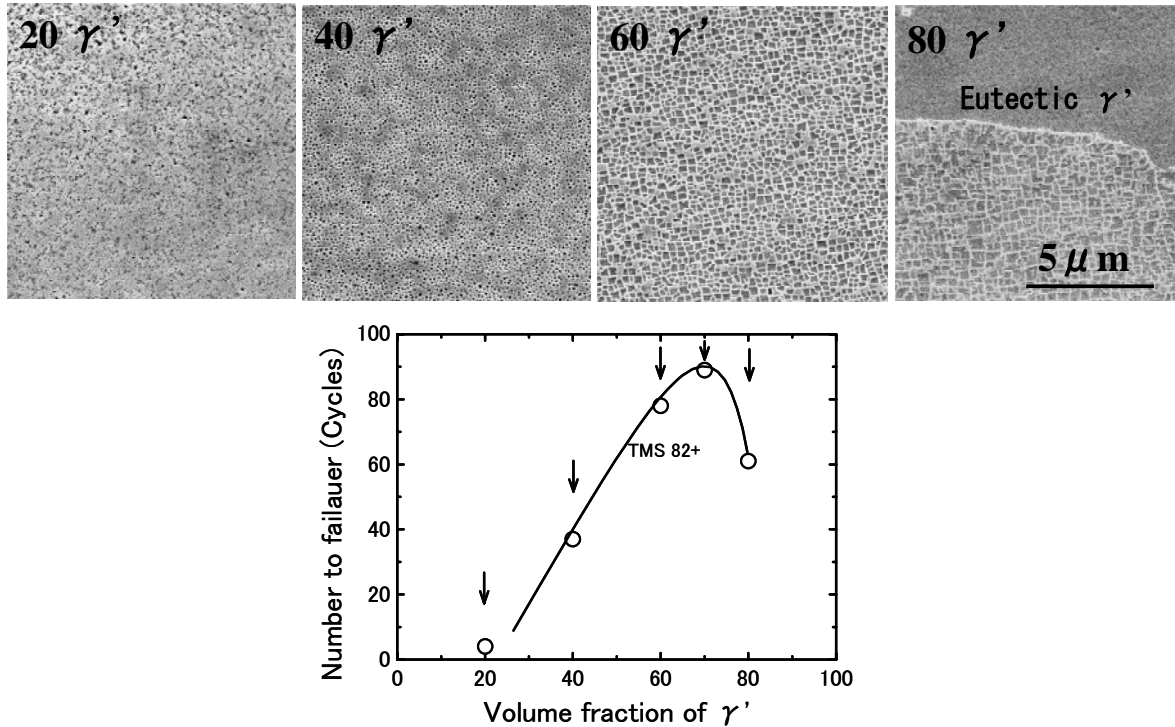


Fig.7 Relationship between γ' volume fraction and cycles to failure, with SEM micrographs of each alloys.

3.2 Effect of γ' volume fraction

The γ' volume fraction is one of the most effective factors affecting mechanical properties. We tested a series of SC alloys designed to be on the 900°C γ/γ' tie-line of alloy TMS-82+, a second generation SC alloy. The TMF results are presented in **Fig.7** with their SEM microstructures. The microstructure changes as designed; the amount of precipitated γ' increases until eutectic $\gamma-\gamma'$ started to deposit. Note that the eutectic $\gamma-\gamma'$ transformed to a single γ' phase during heat treatment. The TMF property improves as the γ' volume fraction increases but decreases as soon as the eutectic $\gamma-\gamma'$ starts to deposit and thus has a peak at about 70 volume % of γ' .

Murakumo and some of the authors [5] reported a same peak of creep strength with tie-line alloys derived from TMS-75, a 3rd generation SC superalloy. These γ' volume fraction dependence of mechanical properties are attributed to a same mechanism, that is, the γ/γ' interface effectively prevents dislocation motions.

3.3 Prediction of TMF property

Based on the findings of compositional and structural effects on TMF properties, regression analysis was carried out to correlate

the stress relaxation with γ' chemical composition and γ' volume fraction. It is shown that the stress relaxation at 900°C is very well expressed as a function of γ' chemical composition and γ' volume fraction of first and second degrees. The result is presented in **Fig. 8**. From the regression coefficients obtained, the most effective compositional factors were found to be Re and Ru. Also the peak strength was calculated to be 74%.

Re and Ru are the elements expected to reduce stacking fault energy in γ phase, as mentioned before, and thus reduce the dislocation mobility especially the climbing of dislocations in γ phase. Considering these, the coefficients obtained in the regression analysis agree with our experimental understandings.

From another regression analysis on 400°C tensile testing data of the same alloys, γ strengthening elements especially Re and W were found most effective in improving the yield strength. An excellent agreement between experimental and predicted yield strengths is presented in **Fig.9**. The higher yield stress obtained by adding Re, W and other strengtheners reduces the area of hysteresis loops and mitigate the microstructure degradation.

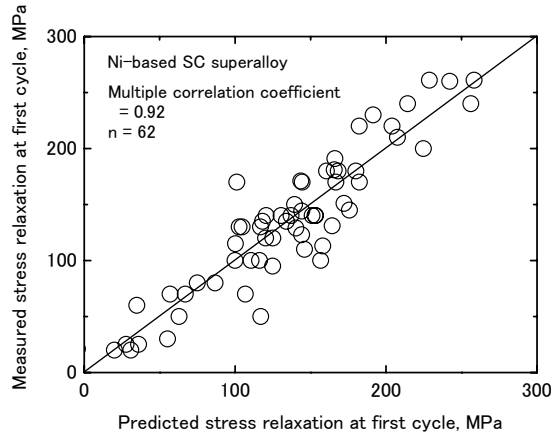


Fig. 8 Relationship between measured and predicted stress relaxation at first cycle. Prediction was made from the chemical composition of γ' and volume fraction of γ' by using regression analysis.

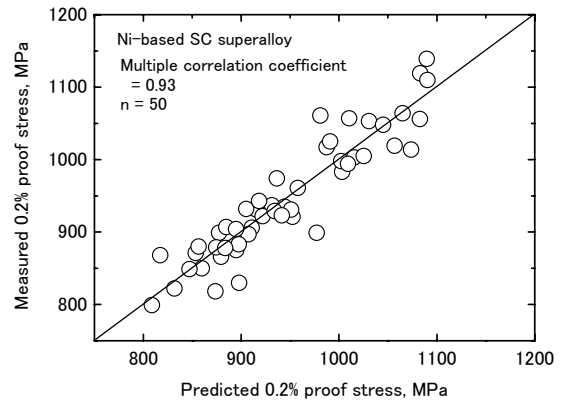


Fig.9 Relation between measured and predicted of proof stress. Prediction was made from the chemical composition of γ' and volume fraction of γ' by using regression analysis.

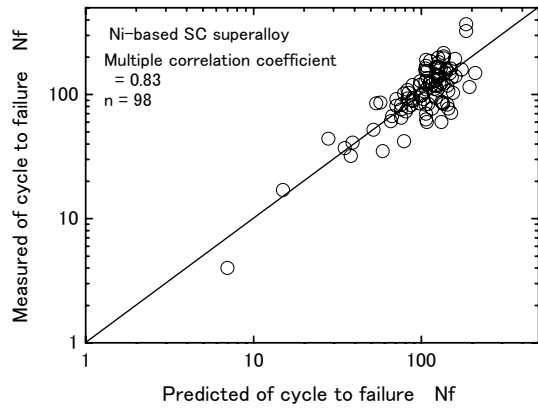


Fig.10 Relationship between predicted cycles to failure from composition of γ' phase and volume fraction of γ' phase and observed cyclic to failure.

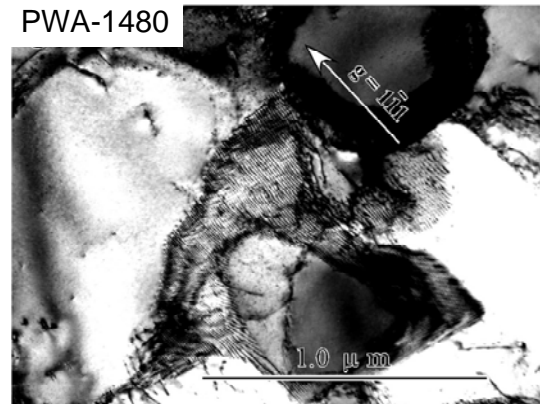


Fig.11 TEM photograph from a TMF specimen of the γ/γ' structure in PWA1480.

A more direct prediction of TMF property, especially cycles to failure, was attempted. All the 98 SC alloy data were used for regression analysis. The cycles to failure was again expressed as a function of the composition and volume fraction of γ' . From the regression coefficients, it was suggested that Re is most effective and Ta comes next. The agreement between experimental and predicted cycles to failure is presented in **Fig.10**. Fairly good agreement has been obtained so far, but it seems that there are possibilities for further improvements in

prediction taking into account some other factors. For example, PWA1480, a 1st generation SC superalloy, exhibited the best TMF property in the 98 tested alloys, the cycles to failure being 368 which is higher than the predicted cycles, 200. **Fig.11** is a TEM micrograph showing a very dense γ/γ' interfacial dislocation network observed in PWA1480 after rupture. We believe this interfacial dislocation network was formed because of that PWA1480 contains highest amount of Ta, 3.8at% (12wt%) , effectively strengthened γ' phase, prevented

dislocation cutting and caused a dislocation pile-up on the interface. The created dislocation network gave a back-stress to the approaching dislocations further to resist the formation of deformation twins [6] and provided the extraordinary strength.

The TMF property, of course, changes depending on the testing condition, e.g., out-of-phase or in-phase or a between, strain, temperature, hold time, etc. Further efforts in this field are very much encouraged to simulate real turbine component operating conditions to select or design right materials to fit into turbines for their higher efficiency and longer durability.

4. Conclusions

Thermo-mechanical fatigue (TMF) property data on 1st to 5th generation single crystal (SC) superalloys generated in NIMS has been analyzed to understand the compositional and structural factors affecting the property.

It was found that Re and Ru additions most effectively improve the out-of-phase TMF property. Ta addition was also found effective in strengthening γ' phase to prevent dislocation cutting and generating dislocation network on the γ / γ' interface, which improves the TMF property. The optimum amount of γ' fraction was found to be about 74%. A prediction equation for the TMF life from a given chemical composition has been derived for selection and design of SC superalloys with better TMS properties.

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