THE AMERICAN SOCIETY OF MECHANICAL ENGINEERS
345 E. 47th St., New York, N.Y. 10017

98-GT-488

A METALLOGRAPHIC TECHNIQUE FOR HIGH TEMPERATURE CREEP DAMAGE ASSESSMENT IN SINGLE CRYSTAL ALLOYS

Pamela Henderson* and Jacek Komenda⁺
*Vattenfall Energisystem AB, P.O. Box 528, 162 16 Stockholm, Sweden
⁺Swedish Institute for Metals Research, Stockholm, Sweden

ABSTRACT

The use of single crystal (SX) nickel-base superalloys will increase in the future with the introduction of SX blades into large gas turbines for base-load electricity production. Prolonged periods of use at high temperatures may cause creep deformation and the assessment of damage can give large financial savings. A number of techniques can be applied for life assessment, *e.g.* calculations based on operational data, non-destructive testing or material interrogation, but because of the uncertainties involved the techniques are often used in combination. This paper describes a material interrogation (metallographic) technique for creep strain assessment in SX alloys.

Creep tests have been performed at 950°C on the SX alloy CMSX-4 and quantitative microstructural studies performed on specimens interrupted at various levels of strain. It was found that the strengthening γ'-particles, initially cuboidal in shape, coalesced to form large plates or rafts normal to the applied stress. The γ-matrix phase also formed plates. CMSX-4 contains ~ 70 vol % γ'-particles and after creep deformation the microstructure turned itself inside out, *i.e.* the gamma "matrix" became the isolated phase surrounded by the γ'-"particles". This can cause problems for computerised image analysis, which in this case, were overcome with the choice of a suitable measurement parameter.

The rafts reached their maximum length before 2% strain, but continued to thicken with increasing strain. Although of different dimensions, the aspect ratios (length/thickness ratio) of the gamma-prime rafts and the gamma plates were similar at similar levels of strain, increasing from ~1 at zero strain to a maximum of ~3 at about 1-2 % strain.

Analysis of microstructural measurements from rafting studies on SX alloys presented in the literature showed that the aspect ratios of the γ- and γ'-phases were similar and that at a temperature of 950-1000°C a maximum length/thickness ratio of about 2.5-3.5 is reached at 1 to 2% creep strain. Measurement of gamma-prime raft or (or gamma plate) dimensions on longitudinal sections of blades is thus a suitable method for high temperature creep damage assessment of SX alloys. This gives a considerable advantage over conventional Ni-base superalloys whose microstructures are usually very stable with respect to increasing creep strain.

INTRODUCTION

The use of single crystal (SX) nickel-base superalloys will increase in the future with the introduction of SX blades into large gas turbines for base-load electricity production. Prolonged periods of use at high temperatures may cause creep deformation and the assessment of damage can give large financial savings. For example, blades exhibiting large amounts of creep strain in localised areas can be taken out of service before failure occurs, so avoiding widespread damage and unplanned outages. Alternatively, it may be found that blades can be run for significantly longer than their original design lives.

In wrought or conventionally cast turbine blades and vanes thermal fatigue cracking is often a cause of premature failure, although creep is an important design criterion. As a first step towards understanding the deformation of an SX alloy which could be used in industrial gas turbines it was decided to study the creep of CMSX-4.

In SX alloys the γ'-particles agglomerate during creep at high temperatures to form large plates (rafts) normal to the applied stress.[1-7] The rafts form in the early stages of creep, gradually increasing in length until a constant raft length is reached.[3,4] A previous study of CMSX-4 showed that while no rafts occurred during creep at 800°C, rafting occurred at low stresses (long times) at 950°C and at all stress levels at 1100°C.[7] In spite of the large number of experiments where rafting has been observed there have been few quantitative microstructural studies of SX alloys reported in the literature. This was the motivation for the work reported here.

**Presented at the International Gas Turbine & Aeroengine Congress & Exhibition
Stockholm, Sweden — June 2–June 5, 1998
This paper has been accepted for publication in the Transactions of the ASME
Discussion of it will be accepted at ASME Headquarters until September 30, 1998**

TABLE 1
Composition in weight % of the CMSX-4 used for creep testing

Crystal. no.	Cr	Co	Mo	W	Ta	Re	Al	Ti	Hf	Ni
V6,V9	6.4	9.7	0.6	6.4	6.5	2.9	5.7	1.0	0.1	Bal
V1,V2,V5	6.5	9.6	0.6	6.4	6.5	2.9	5.6	1.0	0.1	Bal

EXPERIMENTAL

Material and Mechanical Testing

The SX alloy used in the study, CMSX-4, was received in the form of fully heat treated 16 mm diameter bars, having deviations of 3.5 to 12° from the [001] orientation. The bars had been cast and heat-treated by Howmet Exeter Casting, UK. The solution treatment was a multi-step process in which the temperature was raised from 1277°C to 1321°C. The ageing treatment was a two stage process, *viz.* 6h at 1140°C, argon fan quench, 20h at 870°C, air cool. CMSX-4 contains ~70 vol % γ'-particles. The chemical compositions of the master batches from which the crystals were grown are given in Table 1.

Creep testing was performed in air under constant load at 950°C/155 MPa. The testpieces were not allowed to spend more than three hours or less than one hour at the testing temperature before being loaded. The tests were interrupted at specified levels of strain before failure occurred and allowed to cool under load.

Metallography

Creep specimens were sectioned longitudinally along the gauge length and prepared for microstructural examination. One creep specimen (deformed to 8.4% at 950°C) was also examined directly outside the gauge length where the stress (and therefore strain) were lower, but the temperature and creep testing time were the same. The specimens were lightly etched in glyceregia containing 10 ml HNO_3, 20 ml HCl and 20 ml glycerol. Particle shapes and sizes were measured from micrographs taken in a scanning electron microscope (SEM) and quantified by automatic image analysis using a Kontron IBAS 2000 image analysis system. A number of particle parameters were measured, but many were found to be unsuitable. A major drawback to quantifying the γ'-particle / γ-matrix microstructure is that initially, before creep testing, the γ'-particles are discrete and the γ-matrix is the continuous phase. However, after 0.5% creep the matrix is the isolated phase and the γ'-phase is continuous in the form of interlocking rafts. A computerised image analysis system thus measures one γ'-particle as filling an entire measurement frame. The mean chord size was found to be the most suitable parameter for characterising the particles and the matrix. Chord size is defined in Fig. 1.

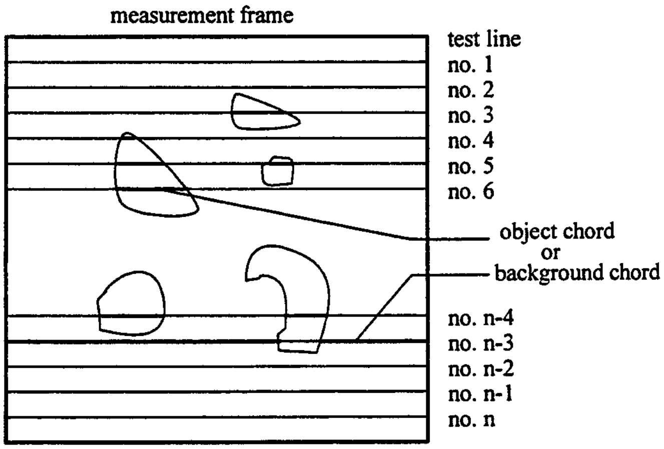


Fig. 1 Definition of chord size. Each measuring frame was divided into 50 tests lines. The length of intersection of the test lines within each particle is called a chord. The mean chord size of all lines within particles is reported. Test lines were produced in the horizontal and vertical directions and the chord size was measured in the matrix and the particles.

RESULTS

Creep Testing

Creep curves are shown in Figs. 2 and 3. There was no primary creep and the creep curve was dominated by the tertiary stage as seen in Fig. 2. Fig. 3 shows, however, that there is a small amount of steady-state creep, about 0.5%, before the start of tertiary. Although small in percentage terms, the steady-state creep stage extends for about 50% of the lifetime.

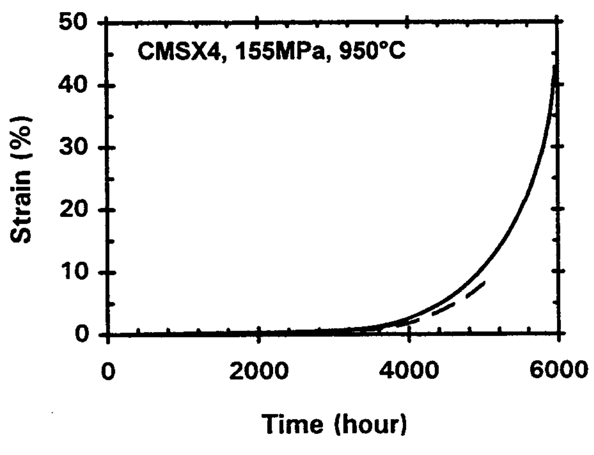


Fig. 2 Creep curves of the investigated specimens. A large amount of tertiary creep.

Microstructural Changes During Creep

The mean values of the particle and matrix dimensions are given in Table 2. Four areas, each of dimensions 29 μm x 29 μm were examined for each specimen. There is a fall in the number of particles measured when rafting begins from 1225 particles at 0% strain to 10 at 0.5% strain, even though the area measured for each specimen is the same. This is because the particles form an interlocking network of rafts and one particle can fill an entire measurement frame. There is also a corresponding increase in the perimeter from 1.6 μm at 0% strain to 1451 μm at 2% strain. The lengths and widths were obtained from chord sizes (see Fig. 1 for an explanation of chord size). With chord measurements a large number can be sampled and this method is therefore more suitable.

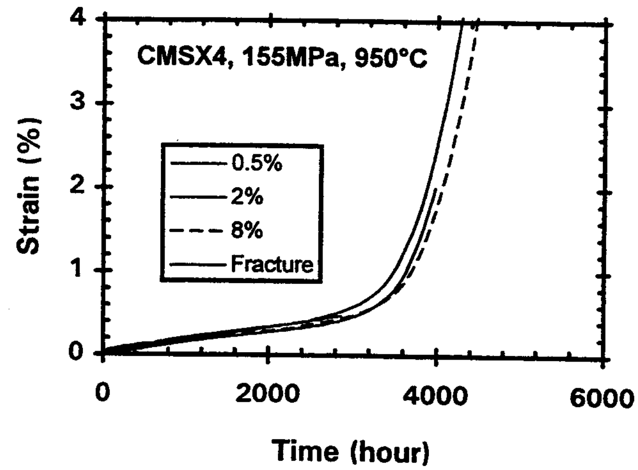


Fig. 3 **(right) Same as Fig. 2, but showing the first part of the creep curves in more detail. A small amount of steady-state creep (~0.5% strain) can be seen.**

TABLE 2

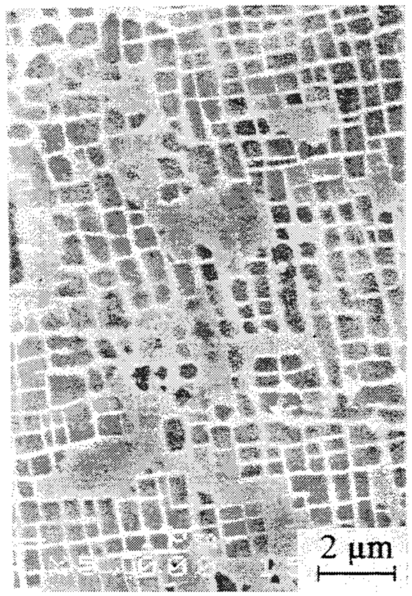
Microstructural measurements taken from longitudinal sections of creep tested specimens. The creep testing conditions were 950°C/155 MPa. Four areas, each of dimensions 29μm x 29μm (a total area of 3364 μm^2) were examined for each specimen. The length is defined as the direction perpendicular ($\perp^r$) to the stress direction and the thickness is the direction parallel ($//^{el}$) to the stress.

Test conditions		Gamma prime particle measurements		Particle/raft dimensions, chord measurements (μm)				Matrix dimensions, chord measurements (μm)			
Specimen no.	Elongation (%)	No.	Perimeter (μm)	No. $\perp^r$	Length $\perp^r$ stress	No $//^{el}$.	Thickness $//^{el}$ stress	No. $\perp^r$	Length $\perp^r$ stress	No. $//^{el}$	Thickness $//^{el}$ stress
V6	0 untested	1225	1.6	2037	0.34 *± 0.004*	2229	0.31 *± 0.004*	2072	0.46 *± 0.016*	2222	0.43 *± 0.016*
V5	0.5	10	335	2532	1.64 *± 0.038*	7622	0.54 *± 0.003*	2441	0.68 *± 0.016*	7523	0.22 *± 0.001*
V2	2.0	6	1451	2263	1.94 *± 0.043*	5945	0.74 *± 0.006*	2159	0.66 *± 0.014*	5828	0.25 *± 0.001*
V1	8.4	5	1365	2425	1.80 *± 0.034*	4241	1.03 *± 0.012*	2329	0.62 *± 0.011*	4144	0.40 *± 0.002*
V1x # (97 MPa)	~0.5%	22	443	2640	1.49 *± 0.038*	6831	0.59 *± 0.004*	2570	0.72 *± 0.016*	6755	0.27 *± 0.001*
V9* rupture	30*	7	805	2515	1.73 *± 0.030*	3072	1.42 *± 0.019*	2422	0.61 *± 0.011*	2972	0.49 *± 0.004*

 # measurements were made immediately outside the gauge length of Spec. V1 between the extensometer ridges and the threaded ends where the stress and therefore strain were lower (denoted Spec. V1x). The stress was 97 MPa and the strain (reduction in area) was about 0.5%.

** the microstructure of the ruptured specimen V9 was quantified away from the fracture at an area of uniform deformation along the gauge length. The strain (reduction in area) was in this case 30% at 950°C. The maximum reduction in area and the elongation of V9 were 46% and 44% respectively.*

The rafts reached their maximum length between 0.5 and 2% strain, but continued to thicken throughout the creep test. Specimen V5, which reached 0.5% strain after 2876 hours showed similar raft dimensions to V1x, which reached ~0.5% strain after 5028 hours. Specimens V1 and V1x both spent the same amount of time at 950°C (5028 h), but experienced different levels of strain and showed different raft dimensions. These two observations show that the raft dimensions depend on the strain rather than time. Fig. 4 shows the original microstructure and after rafting at 0.5% strain. The aspect ratios (length/thickness) of the γ' and the γ phases are plotted as a function of strain in Fig. 5. It can be seen that they are similar, reaching a maximum at 0.5-1.0 % strain.



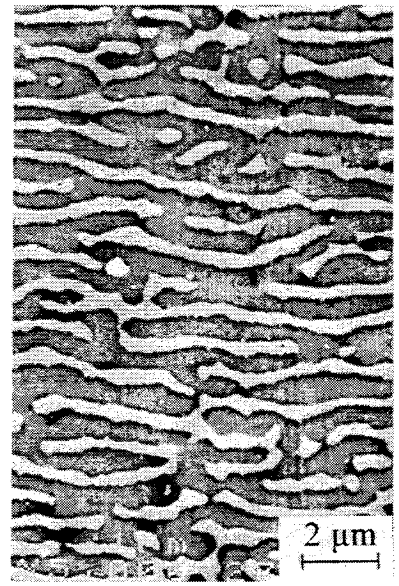


Fig. 4 **(Left) Original microstructure before creep testing. (right) Microstructure after 0.5% strain. Secondary electron image. Stress direction vertical. γ' is the dark phase. Before creep the γ-matrix is the continuous phase, but after creep the γ'-phase is continuous.**

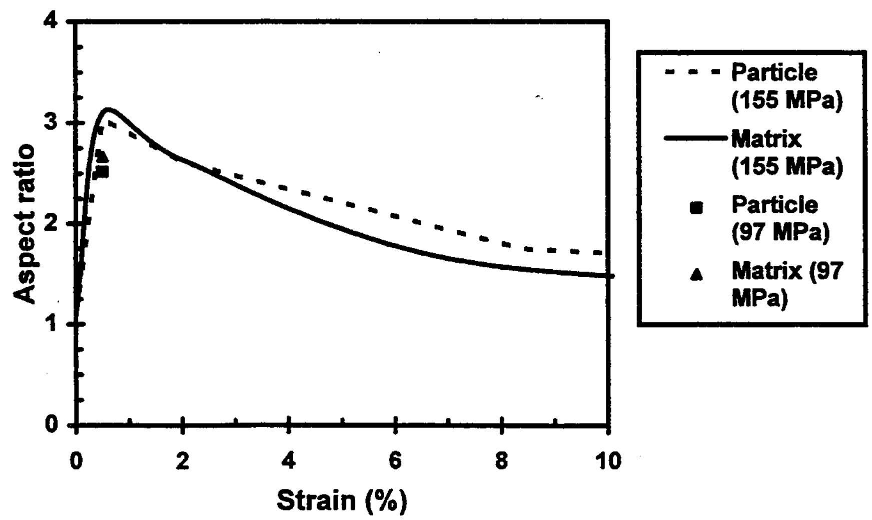


Fig. 5 **The aspect ratio (length/thickness ratio) of γ'-particles and the γ-matrix as a function of creep strain at 950°C and 155 MPa.**

DISCUSSION

Measurements of γ'-rafting in an SX alloy at 982°C have been presented in Ref. 3 and rafted microstructures from an interrupted creep test on SC16 at 950°C have been presented in Ref. 15. The aspect ratios of the γ'-particles during creep at 982°C were calculated from length, thickness, time and strain information presented in Ref. 3 and replotted as aspect ratio versus strain in Fig. 6. The aspect ratios of the γ and γ' phases from Ref. 15 were measured by chord measurements on micrographs presented in

Ref. 15 and the results are shown in Fig. 7. They show a similar trend to the aspect ratio results reported here (see Fig. 5) although the maximum aspect ratio is reached at a slightly higher level of strain. In Fig. 7, 2% strain was reached after only 238 h, whereas in Fig. 5 0.5% and 2% strain were reached after 2876 h and 3978 h respectively.

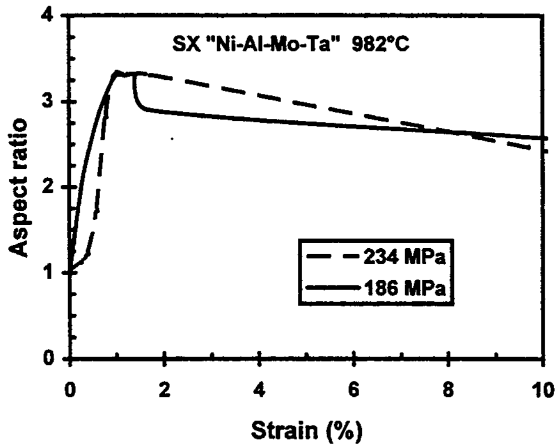


Fig. 6 The aspect ratio (length/thickness ratio) of γ′-particles versus creep strain for an SX alloy tested at 982°C. The aspect ratios have been calculated from length and thickness versus time information and from time-strain creep curves presented in Ref. 3.

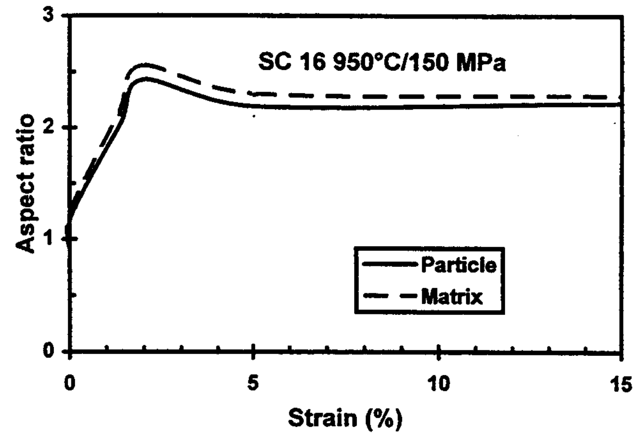


Fig. 7 The aspect ratios of particles and matrix measured from micrographs presented in Ref. 15. SC16 contains ~40 vol % γ′-particles.

As rafting is a process which depends on diffusion and needs a finite amount of time to occur, it is likely that the maximum aspect ratio will be shifted to higher strain levels if the strain rate is high. It is known, for example, that rafting does not occur during conventional high temperature tensile tests on SX alloys. In industrial gas turbines the strain rate is slow and so rafting will be strain-controlled.

The use of microstructural changes to predict the remanent life of nickel-base superalloys is less advanced than in ferritic alloys. In wrought Ni-base alloys grain boundary cavitation occurs in much the same way as in ferritic steels.[8] In cast alloys the situation is more complex; cavities can occur and rafting may occur to a limited extent.[8,9] Particle growth, precipitation of carbides and the precipitation of topologically close packed phases like σ also occurs.[8-11] In some cases the most noticeable change in the microstructure was the increase in dislocation density and this required the use of transmission electron microscopy.[12,13]

SX alloys tolerate higher temperatures than conventional Ni-base superalloys and rafting is more likely to occur because :-

(a) the magnitude of the γ/γ' misfit is larger (more negative) at higher temperatures,

(b) in commonly used <001> crystals all the γ'- particles are oriented with some faces normal to the stress and

(c) in modern SX alloys the γ' volume fraction is 0.5-0.7 which means that particle impingement is easier.

Although the rafting process is not totally understood it is thought that plastic strain is a prerequisite for rafting.[6, 14] The results from this study show that the raft dimensions depend on the amount of creep strain and thus it seems that high temperature creep damage will be easier to assess in SX alloys than in their conventionally cast counterparts. It must be stressed that no rafts have been seen after low temperature (750~800°C) creep of CMSX-4 [7,16] and other methods will have to be developed to estimate creep damage at these temperatures. Preliminary results indicate that rafting occurs in low temperature creep strained material after a suitable post-creep high temperature heat treatment[17].

CONCLUSIONS

Creep tests have been performed on the single crystal alloy CMSX-4 at 950°C and the changes occurring in the γ' particles have been quantified, to provide a basis for creep damage assessment of single crystal turbine components.

Before creep testing, the γ'-particles (~70 vol %) were cuboidal and discrete and the γ-matrix was the continuous phase. However, after 0.5% creep the "matrix" became the isolated phase and the γ'-phase formed a continuous network of interlocking rafts. The rafts reached their maximum length before 2% strain, but continued to thicken throughout the test.

Chord length measurements were found to be the most suitable method of quantifying the microstructure by automatic image analysis, because they can be used for discrete and continuous phases.

Although of different dimensions, the aspect ratios (length/thickness ratios) of the γ' rafts and the γ-matrix plates were similar at the same levels of strain, increasing from 1 at zero strain to a maximum of 3 at about 1% strain. Analysis of rafting data presented in the literature showed that this was also true for other SX alloys provided that enough time is allowed for rafting to occur.

Measurement of γ' raft (or matrix plate) aspect ratios on longitudinal sections of SX turbine blades is a method suitable for creep strain assessment.

ACKNOWLEDGEMENTS

The creep testing and microscopy were performed at the Swedish Institute for Metals Research. We thank Joakim Lindblom and Facredin Seitisleam for experimental help and Leif Berglin (ABB STAL, Finspång, Sweden) for useful discussions. The research was entirely funded by *Värmeforsk* (The Swedish Thermal Power Research Foundation) and additional financing was provided by *Vattenfall Värmekraft AB* (Vattenfall Thermal Power). The financial support is gratefully acknowledged.

REFERENCES

1. D.D. PEARSON, F.D. LEMKEY and B.H. KEAR: Stress Coarsening of γ' and its Influence on Creep Properties of a Single Crystal Superalloy. *Proc. 4th Int. Symp. on Superalloys*. Eds. J.K. Tien *et al.*, pp 513-519, ASM Ohio, 1980.
2. R.A. MacKAY and L.J. EBERT: The Development of Directional Coarsening of the γ'Precipitate in Superalloy Single Crystals, *Scripta Metall.*, 1983, **17**, pp 1217-1222.
3. R.A. MacKAY and L.J. EBERT: The Development of γ- γ' Lamellar Structures in an Nickel-Base Superalloy during Elevated Temperature Mechanical Testing, *Metall. Trans. A*, 1985, **16A**, pp 1969-1982.
4. M.V. NATHAL and L.J. EBERT: Gamma Prime Shape Changes During Creep of a Nickel Base Superalloy, *Scripta Metall.*, 1983, **17**, pp 1151-1154.
5. T.M. POLLOCK and A.S. ARGON: Directional Coarsening in Nickel-Base Single Crystals with High Volume Fractions of Coherent Precipitates, *Acta Metall. Mater.*, 1994, **42**, pp 1859-1874.
6. M.V. NATHAL and R.A. MACKAY: The Stability of Lamellar γ-γ' Structures, *Mater. Sci and Eng.*, 1987, **85**, pp 127-138.
7. H. MUGHRABI, W. SCHNEIDER, V. SASS and C. LANG: The Efffect of Raft Formation on the High Temperature Creep Deformation Behaviour of the Monocrystalline Nickel-Base Superalloy CMSX-4, *Proc. ICSMA-10* Eds Oikawa *et al.* pp705-708. JIM, Sendai, 1994.
8. S-Å KARLSSON, C PERSSON and P-O PERSSON: Metallographic Approach to Turbine Blade Lifetime Prediction. BALTICA III Int. Conf. on Plant Condition and Life Management, pp 333-349, Eds.S. Hietanen and P Auerkari, VTT, Espoo, Finland, (1995).
9. C. PERSSON and P-O PERSSON: Evaluation of Service Induced Damage and Restoration of Cast Turbine Blades. Proc Conf. " Superalloys 1992", pp 867-876, Eds. S.D. Antolovich *et al.* TMS, (1992).
10. R.CASTILLO, A.K. KOUL and J-P. A. IMMARIGEOM: The Effect of Service Exposure on the Creep Properties of Cast IN 738 LC . Superalloys 1988, pp805-814, Eds S. Reichman et al. TMS (1988).
11. A.K. KOUL and R. CASTILLO: Assessment of Service Induced Microstructural Damage and its Rejuvenation in Turbine Blades. Met. Trans. **19A**, pp 2049-2066, (1988).
12. P.J. HENDERSON and M. McLEAN: Microstructural Changes During the Creep of a Directionally Solidified Nickel-base Superalloy. Scripta Metall., **19**, pp 99-104, (1985).
13. P.J. HENDERSON: Dislocations at γ/γ' Interfaces during the creep of Nickel-base Superalloys. Scripta Metall., **22**, pp1103-1107, (1988).
14. M. VÉRON and P. BASTIE: Strain Induced Directional Coarsening in Nickel Based Superalloys. Acta Mater., **45**, pp 3277-3282, (1997).
15. D. MUKHERJI, H. GABRISCH, W. CHEN, H.J. FECHT and R.P. WAHI: Mechanical Behaviour and Microstructural Evolution in the Single Crystal Superalloy SC16. Acta Metall., **45**, pp 3143-3154, (1997).
16. P. J. HENDERSON and J. KOMENDA: Changes in the Microstructure During the Creep of a Single Crystal Alloy. Proc. Conf. Advances in Turbine Materials, Design and Engineering. pp 663 - 678, Eds A. Strang *et al.* The Institute of Materials, London, UK, (1997).
17. C. JANSSON, L. BERGLIN and P. J. HENDERSON. Creep Damage in a Single Crystal Turbine Blade Alloy. Swedish Thermal Power Research Association, *(Värmeforsk)*. M6-626, in press, (1998).