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The Analysis of Low Cycle Fatigue Behavior in a Nickel Based Superalloy

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ABSTRACT

The deformation behaviour and damage mechanisms of the wrought nickel based superalloy EI 698 VD has been investigated under creep/fatigue loading conditions. The alloy was subjected to a load controlled isothermal low cycle fatigue at 650°C with superimposed hold periods of constant tensile load simulating creep stress components. The introduced hold periods were in the range of 1 minute to 10 hours. The pure fatigue and pure creep tests were conducted as well. The tests were of tension–tension type defined by stress ratio R = 0.027. During hold periods a constant stress of σ = 740 MPa was maintained corresponding to creep loading. The tests were conducted until fracture. The influence of hold time was evaluated through the deformation behaviour, fatigue life, and fracture mechanisms participating in the failure process. The time to fracture decreased continuously with increasing hold period. Considering the fatigue life achieved after shortening the hold time to 1 minute, the life time measured was more than 10 times higher than in the case of pure creep. The strain rate dependence achieved within this testing showed inverse dependence with respect to the hold time. The microstructure examination of the fractured specimens, using transmission electron microscopy, made it possible to reveal the participating dislocation mechanisms for combined loading. The fracture analysis of broken up specimens showed that fatigue mechanisms participated in crack nucleation and crack propagation of of testing time when the hold time was 30 minutes and less.

Keywords : Nickel superalloy, low cycle fatigue, tensile hold time, fatigue life, microstructure analysis, fracture analysis.

INTRODUCTION

Many components in high temperature applications are subjected to loading that requires resistance to creep. In many cases, it was recognised that the static creep and/or conventional fatigue test conditions approach cannot always assess the deformation behaviour and life of the component. For example, various components of aircraft jet engines experience periods of both fluctuating and steady stress, due to the complex situation of mechanical and thermal stresses originating from centrifugal force, high frequency vibrations and temperature transients as the aircraft flies (Harkgard and Gueden, 1988).

Permanently increasing attention has been paid to the study of the creep and fatigue interaction in either isothermal or anisothermal fatigue conditions (Wangyao, *et al.* 1997; Zrnik, *et al.* 2001; Zrnik, *et al.* 1997; Zrnik, *et al.* 1998; and Zrnik, *et al.* 1997). In the past two decades considerable effort has been brought to characterise the deformation process of nickel based superalloys that were stressed under the conditions of time-dependent load at elevated temperatures (Härkgard, *et al.* 1988; Wangyao, *et al.* 1997; Zrnik, *et al.* 2001; and Zrnik, *et al.* 1997;). In such cases both creep and fatigue can contribute to the degradation of the material. The introduction of hold periods in a low cycle fatigue test at high temperature can be considered as a frequency effect and/or as an effect of the time dependence process, and is the widely used method of studying creep-fatigue interaction in high temperature alloys. The introduction of tensile hold periods during the LCF test has been shown to result mostly in a decrease in the number of cycles to failure relative to continuous cycling (Leveillant, *et al.* 1977; Zrnik, *et al.* 1998;

and Chateau, *et al.* 1998). Rarely beneficial effects of LCF with hold periods resulting in increasing fatigue was reported for nickel based superalloys (Nardone, *et al.* 1983; Antalovich, *et al.* 1981; and Tien, *et al.* 1989). Deformation characteristics under the creep-fatigue stress can differ considerably from those of the static creep.

In the present investigation we have studied the deformation behaviour and the cyclic life of EI 698 VD wrought polycrystalline nickel based superalloy subjected to creep-fatigue loading. In load controlled cyclic creep tests the hold periods introduced at tensile amplitude peaks were variable and their influence has been assessed through deformation characteristics, whereas the stress range interval was kept constant. The development of the deformation process and damage mechanisms in dependence of the applied load schedules was evaluated using TEM of thin foils and SEM of fractured surfaces.

EXPERIMENTAL

The wrought nickel based superalloy EI 698 VD was selected as an experimental material. This alloy is suitable for the manufacturing of discs and shafts of aircraft engines operated at temperatures of up to 750°C. The chemical composition of the alloy in mass % is as follows: C max. 0.08, Cr 13-16, Mo 2.3-3.8, Nb 1.8-2.2, Ti 2.3-2.7, Al 1.3-1.7, Fe max. 0.2, balance Ni. The microstructure of superalloy was a result of heat treatment and consists of the equiaxed grain structure, Figure 1. The alloyed nickel FCC matrix is strengthened by coherent gamma prime precipitates, Figure 2. The carbides of MC and $M_{23}C_6$ types that do not contribute substantially to the matrix strengthening but stabilize and strengthen the grain boundaries were present in the alloy.

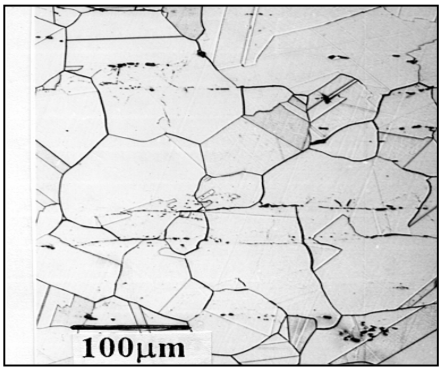

Figure 1 Micrograph of alloy structure

The tensile stress cycling load controlled tests were conducted at a temperature of 650°C. The cyclic creep tests were of a trapezoidal wave pattern. The seven different hold times $\Delta t = 0$ (pure fatigue), 1, 3, 7.5, 15, 30 minutes and $\Delta t = 1$, 3, 5 and 10 hours at peak stress $\sigma = 740$ MPa were introduced in the tensile part of the load cycle. The net effect of these hold times is to systematically impose a creep stress component on the fatigue load cycling. The cycling frequency range was between 5.5×10^{-3} and 2.7×10^{-5} Hz and stress ratio R = 0.027. The stress ramp rate in one cycle, either during on-load or the off-load period, was 7.4 kN/min. No hold time was maintained at a reduced load level of 20 MPa. The specimen longitudinal deformation, the fracture lifetime or total time of the cyclic test, the number of cycles to fracture, and the time at maximum load during cyclic test were recorded and compared with static creep.

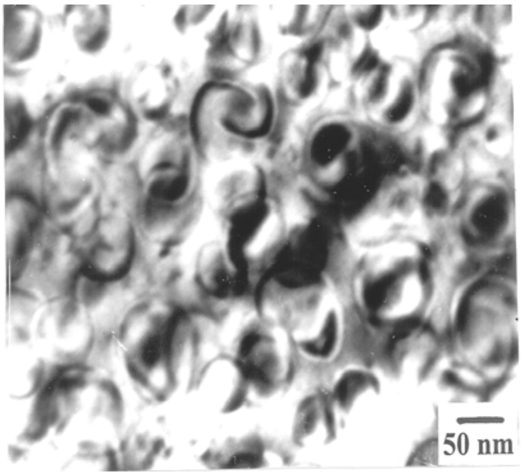

Figure 2 Morphology of gamma prime

The microstructure characteristics of fractured specimens were studied by transmission electron microscopy (TEM) of thin foils prepared from the gauge sections. The fracture surfaces were examined using scanning electron microscopy (SEM) with an aim to evaluate the participation of the fatigue stress component in crack initiation process.

RESULTS AND DISCUSSION

The strain – time to failure dependencies, measured when strain was at the maximum load, corresponding to initial stress of 740 MPa, for isothermal cyclic creep tests for longer hold periods are presented in Figure 3. The longer hold periods varied between 1 hour to 10 hours. The strain-time when shorter hold periods in the range of 1 minute to 30 minutes were applied are presented in Figure 4. Comparing the results of the cyclic creep with that of pure creep data the introduction of any hold period in cycling resulted in fracture life increase and a decrease in creep strain rate ε. There is only a slight scattering of ε values observed for 10, 5, 3, 1 and 0.5 hours hold periods respectively. However, the introduction of the shorter hold periods caused the creep strain to drop down to more than half compared with that of pure creep.

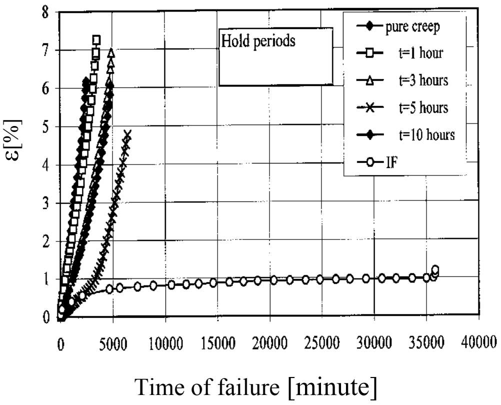

Figure 3 Strain–time to failure dependencies.

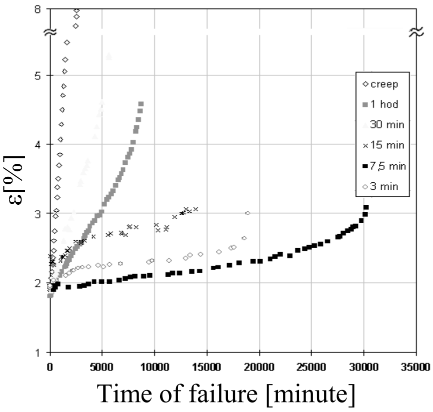

Figure 4 Strain-time to failure dependencies

The results on the total time to failure (TTF), the time corresponding to maximum (creep) load (MLT), numbers of cycles to failure (NCF), and fracture strain received at the cyclic creep experiment for shorter hold periods are summarised in Table 1.

A plot of the number of cycles N_f to failure vs. hold time Δt, which is shown in Figure 5 indicates a sharp transient lifetime at hold periods between 7.5 minutes to 15 minutes. This figure also includes the N_f value obtained from a pure low cycle fatigue test conducted without hold periods using the same fatigue stress cycle parameters.

Table 1 Experimental data received at cyclic test

Parameter	**Hold time [minute]**							
	fatigue	**creep**	**1**	**3**	**7.5**	**15**	**30**	**60**
TTF [min]	44 268	2 500	54 972	18 942	30 300	13 896	5 662	3 406
MLT [min]	-	2 500	10 741	8 003	19 591	10 911	4 981	3 188
NCF [min]	22 120	-	10 778	2 668	2 612	728	166	53
ε_f	3.2	6.3	3.3	3.5	3.6	3.1	3.9	6.9

In order to evaluate the creep fatigue resistance of tested nickel based superalloy the time criteria, such as time to failure or time to failure corresponding only to the maximum applied load can be used for this purpose. The evaluation of deformation behaviour according to the time to failure corresponding to the sum of hold periods at maximum load (MLT) is presented in Figure 6. The corresponding hold period of Δt = 7.5 minutes at maximum load seems to have specific influence on the deformation behaviour of the alloy. Probably, in the cyclic creep with the hold time shorter than 7.5 minutes, in damage process more fatigue would participate at crack nucleation and its propagation. If hold time is over this critical dwell the life prediction dominating role in the damage process would be taken over by creep. When comparing these results with the results received on total time to fracture, a contradiction appeared. The longest life was corresponding to the test with a hold time of Δt =1 minute. In the case that the total number of cycles to fracture was the criterion to evaluate the deformation behaviour of the alloy the plot representing the dependence is documented in Figure 7.

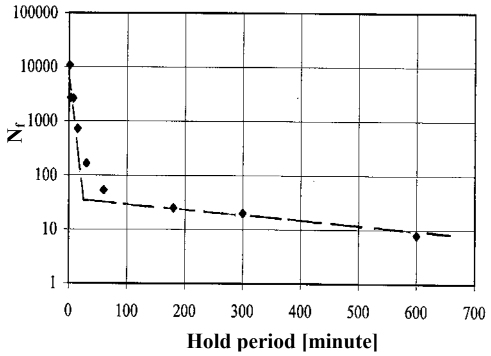

Figure 5 Numbers of cycles to failure in dependence of hold time.

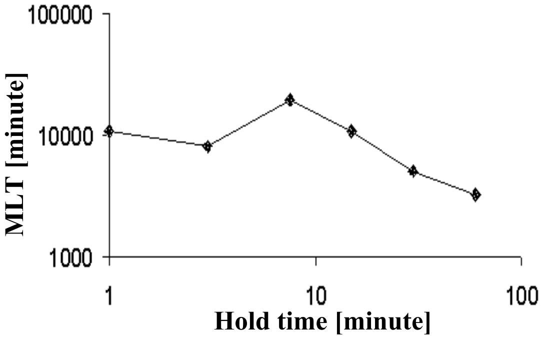

Figure 6 Plot of time to failure corresponding to sum of hold time at maximum load.

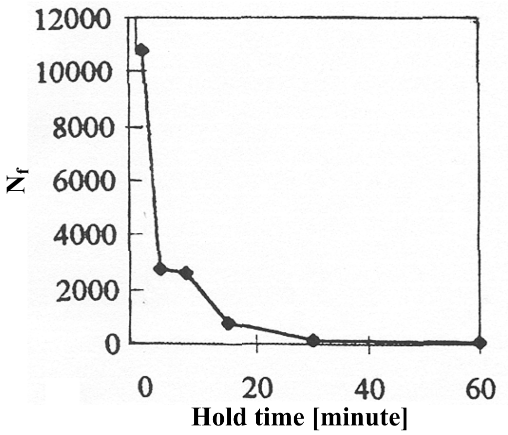

Figure 7 The dependence of the hold time and the number of cycles to failure.

Regardless of the fact that there is an observed continuous decrease in the number of cycles to failure with increasing hold time, Δt the relationship can not be interpreted generally as a prior creep damage effect on the fatigue mechanisms and/or as influence of creep on cycles reduction. The main reason not to follow such interpretation is the fact that creep damage which is a time-controlled process will simply dominate at longer hold times. That is why it would therefore be illogical to explain such behaviour to apply the concept of the major damage mechanism to influence the minor one. Besides that, the resulted number of cycles to failure which showed continuous decreasing tendency with increasing hold time, just that it does not need to be the result of creep fatigue interaction. It can be accepted only as a pure mathematical relation between time to failure and corresponding cycle number causing the creep damage. Besides that, the resulting number of cycles to failure which showed continuous decreasing tendency with increasing hold time just it does not need to be the result of creep fatigue interaction. It can be accepted only as a pure mathematical relation between time to failure and corresponding cycle number causing the creep damage.

Structural Analysis

An examination of the deformation dislocation microstructure developed in the fractured specimens due to variation of hold periods revealed the following characteristic features.

1. Any coarsening and coalescence of gamma prime precipitates was not observed.
2. The dislocation arrangement was homogeneous in matrix and dislocations produced dense dislocation networks under only creep testing. The dislocation – gamma prime particle interaction provided the evidence that Orowan bowing mechanism, Figure 8, and particle shearing mechanism, Figure 9, participated in dislocation surpassing the gamma prime particles.

In the matrix at the early stage of deformation only narrow dislocation slip bands were present in the matrix in specimens subjected to pure fatigue, Figure 10. In the advanced stages of cyclic deformation more slip bands in different slip systems were formed and dislocation tangling among slip bands was observed. Within the slip band, particle shearing appeared to be active in surpassing the gamma prime precipitates, Figure 11. Such dislocation arrangement can result in an extensive hardening causing a decrease in ductility. The similar dislocation arrangement was observed in specimens subjected to cyclic creep with shorter hold periods of up to 15 minutes.

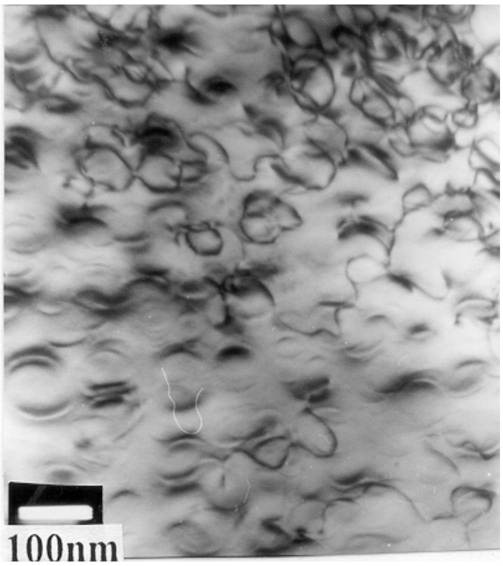

Figure 8 TEM micrograph of Orowan dislocation bowing passing particles

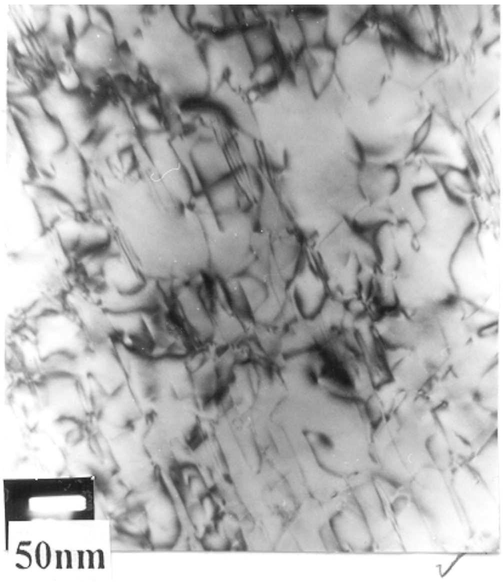

Figure 9 TEM micrograph of particle shearing.

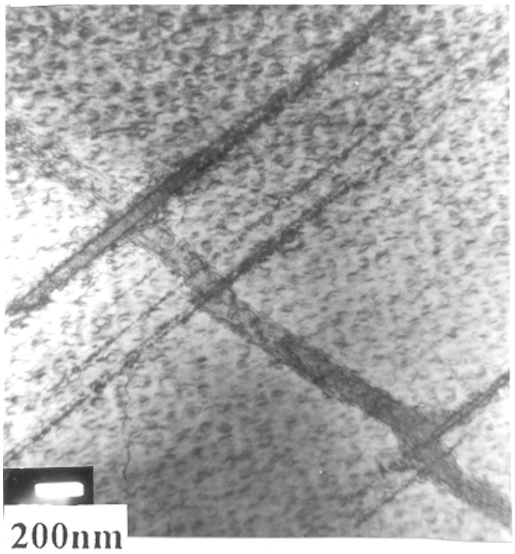

Figure 10 Multiple slip bands. Pure fatigue.

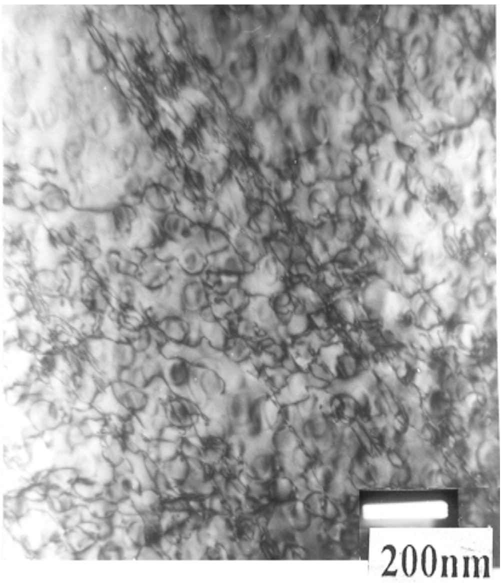

Figure 11 Dislocation tangling and particle shearing within slip band.

However, as hold time was prolonged, the more creep stress component participated in cyclic deformation process and dislocation structure resulting from these types of loading regimes of cyclic creep indicates a mode of deformation which is quite similar to that of pure creep. Regardless of the different hold periods over 30 minutes the dislocation structure in fractured specimens bore the features of severe plastic deformation and the dense dislocation clusters and tangles were found homogeneously distributed in the matrix.

On the basis of microstructure observation we can explain the observed deformation behaviour of the alloy and/or we can relate the achieved strain characteristics at mechanical testing with participating dislocation deformation mode in the time of the different cyclic creep loading. In the creep-fatigue deformation process the strain contribution rising at initial loading (on-load) of each new cycle was not observed. This indicates the strain rate would result only in duration of hold period, i.e. of acting creep stress. This effect was observed only if hold time was over 15 minutes, including. If more frequent load reduction was involved in testing it was more difficult to recognise what part of the loading cycle contributed more substantially to deformation, either immediate effect of initial on-load period or hold time period within cycle.

On the basis of deformation microstructure analysis and analysing fracture mode it is possible to state that decisive or significant influence in deformation process, when shorter hold periods were introduced, was due to contribution from the cyclic strengthening. The material softening is more effective when longer hold periods are introduced. This effect is cycle dependent and therefore it can be considered to be a fatigue driven process.

Fracture Analysis

The SEM fracture analysis of broken specimens was employed to trace the crack tip initiation site and its morphology, as well as the crack propagation mode with consideration to microstructure. Structure investigation along the plane normal to the crack propagation revealed that the crack initiation took place dominantly at the intersections of grain boundaries with the specimen surface regardless of the hold time. Secondary cracks, which were few on cross section, prevailingly nucleated along grain boundaries perpendicular to the applied stress and had either wedge or flat morphology. All fractures formed either at creep or at cyclic-creep with long hold periods had characteristic intergranular crack initiation and propagation mode. The typical morphology of fracture surface with characteristic intergranular features is shown on Figure 12.

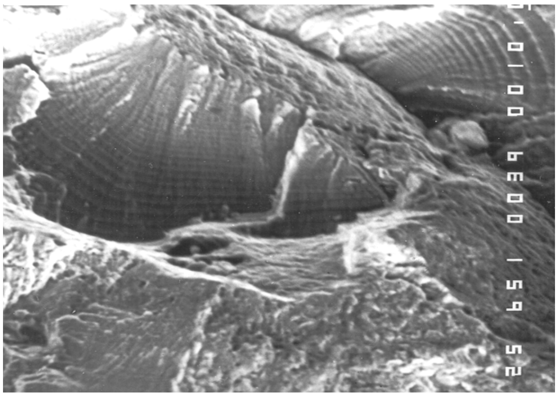

Figure 12 Micrograph of intergranular fracture

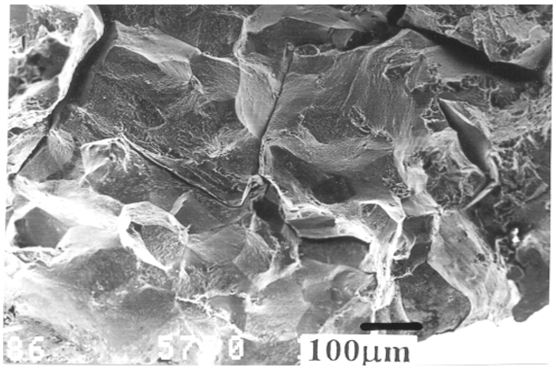

Figure 13 Crack initiation by fatigue

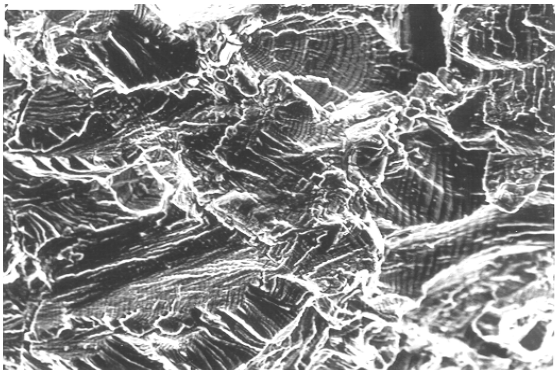

Figure 14 Intragranular fatigue cleavage

On the basis of fracture mechanism investigation at the damage process, regardless of the hold time period, the dominant role of intergranular fracture was clear. This fact proves the decisive contribution to deformation process belonged to creep. However, this conclusion is conceiving and cannot be accepted as only a proof that fatigue was absent in damage. It was found and verified that at fatigue testing defined by a small amplitude without hold time, the intergranular cleavage dominated. From the fractography analysis it was evident that participation of fatigue mechanisms in the damage process appeared first time when a hold time of 30 minutes and shorter was introduced in to low cycle fatigue. In these cases the intragranular crack initiation of fatigue origins were present, Figure 13. Later in the advanced stages of fracture the crack proceeded by intergranular mechanisms. The effect of fatigue was more pronounced as hold time became shorter. The intragranular fracture with characteristic fatigue striation on the fracture surface for a hold time of 3 minutes is documented in Figure 14.

Very of often after critical crack opening, the further propagation of the crack was of mixed fracture mode, intergranular and intragranular cleavage. As hold period was cut down more intragranular facets with fatigue striations appeared on fracture surfaces. To relate results on mechanical testing with fractography results the conclusion can be made that if fatigue dominates the deformation process a substructure of narrow dislocation shear bands was observed and dislocation slip was more localised, which was reflected in higher strengthening.

CONCLUSIONS

The mechanisms of deformation and damage at high temperature in wrought nickel based superalloy were investigated under creep and isothermal cyclic creep. The following conclusions can be drawn from the present study:

1) The creep-fatigue interaction represented by tensile hold period introduction onto creep stress showed no detrimental effect in creep life no matter what periods of holds have been used

2) Introduction of the tensile hold period has been shown to result in a decrease in the number of cycles to failure as the hold period was prolonged. The reduction in the strain rate at isothermal cyclic creep was observed. The more pronounced strain rate reduction can be related to a more active fatigue participation in the deformation process.

3) The creep-fatigue interaction was reflected in a change of deformation process mode. Whereas the creep process is characterised by homogeneous dislocation arrangements, with prevailing cycling stress, mainly in the initial stages of testing, the deformation is more inhomogeneous and condensed in more narrow slip bands. Orowan bowing and particle shearing mechanisms were active regardless of the hold periods.

4) The presence of fatigue stress participation in the deformation process dominated when the hold period was below 0.5 hour.

REFERENCES

Antalovich, C. D., Liu, S., and Baur, R. 1981. *Met. Trans. A.* **12A**. : 473.

Chateau, E., Remy, L., Dolet-Berge, N., and Fournier, D. 1998. *Proceedings of the 6th Liege Conference on Materials for Advanced Power Engineering 1988, Liege, Belgium, September 1998, Schriften des Forschungszentrum Jülich.* : 1197.

Härkgard, G. and Guėdon, J. G. 1998. *Proceedings of the 6th Liege Conference on Materials for Advanced Power Engineering 1988, Liege, Belgium September 1998, Schriften des Forschungszentrum Jülich.* : 913.

Leveillant, C., Rezgui, C., and Pineau, A. 1977. *Mechanical Behaviour of Materials.* Oxford, Pergamon Press. : 163.

Nardone. V. C., Matejczyk, D. E. and Tien, J. K. 1983. *Met. Trans A.* **14A** : 1435.

Tien, J. K., Nair, S. V. and Nardone, V. C. 1989. *Superalloys, Supercomposites and Superceramics*. New York, Academia Press. : 301.

Wangyao, P., Nisaratanaporn, E., Zrnik, J., Vrchovinsky, V. and Hornak, P.,1997. High temperature properties of wrought nickel base superalloy in creep-fatigue conditions. *J. Met. Mater. Miner.* **7(1)** 1-12.

Zrnik, J., Semenak, J., Wangyao,P., Vrchovinsky, V. and Hornak, P. 2001. Structure and fotography analysis of nickel base superalloy subjected to low cycle fatigue. *Materials Structsture and Micromechanics of Fracture. (MSMF-3),* Czech Republic. : 669-676.

Zrnik, J., Wangyao, P., Vrchovinsky, V. and Hornak, P. 1997. Thermomechanical fatigue of wrought nickel base superalloy. *5th European Conference on Advanced Materials and Process (EUROMAT97)* , Netherland. *:* 181-184.

Zrnik, J., Wangyao, P., Vrchovinsky, V., Hornak, P. and Nisaratanaporn, E. 1998. Deformation and damage mechanism of nickel base superalloy subjected to creep, isothermal cycle creep and thermomechanical fatigue. *10th International Symposium, Metallography'98,* Slovakia. *: 242-246.*

Zrnik, J., Wangyao, P., Vrchovinsky, V., Hornak, P. and Mamuzic, I. 1997. Deformation behavior of wrought nickel base superalloy in conditions of thermomechanical fatigue. *J. Metall.* **36(4)** : 225-228.

Zrnik, J., Wangyao, P., Vrchovinsky, V., Hornak, P. and Nisaratanaporn, E. 1998. Deformation behavior of wrought nickel base superalloy subjected to isothermal cycle creep and thermomechanical fatigue. *4th International Conference on Low Cycle Fatigue and Elasto-plastic Behavior of aterials*, Germany : 124.

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