RARE METALS
Vol. 30, Spec. Issue, Mar 2011, p. 477
DOI: 10.1007/s12598-011-0328-z

Microstructural evolution of a directionally solidified nickel-base superalloy during thermal fatigue

XIA Pengcheng[a,b], YANG Lei[a], YU Jinjiang[b], SUN Xiaofeng[b], GUAN Hengrong[b], and HU Zhuangqi[b]

[a] *School of Materials Science and Engineering, Shandong University of Science and Technology, Qingdao 266510, China*
[b] *Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016, China*

Received 29 September 2010; received in revised form 29 December 2010; accepted 10 January 2011
© The Nonferrous Metals Society of China and Springer-Verlag Berlin Heidelberg 2011

Abstract

The transition of γ' phase and dislocation during thermal fatigue test was investigated. There is γ' denuded region in two sides and the tip of thermal fatigue crack because of oxidation during thermal fatigue test. γ' phase coarsens and rafts. The direction of γ' rafting is perpendicular to the orientation of crystal growth. High density dislocations are found near crack. Single dislocation moves in the matrix apart from crack. A few dislocations shear γ' phase because of relatively low strength of γ' phase at high temperature.

Keywords: nickel-base superalloy; thermal fatigue; γ' rafting; dislocations

1. Introduction

Superalloys are widely used in a variety of applications at the condition of aggressive atmospheres such as those experienced in gas turbine, jet engine etc [1-2]. They works for a long time at 650 – 1200 °C and sophisticated stress. Thermal fatigue is a potential mode of failure in many components of superalloys such as blades, vanes etc, which are exposed to fluctuating temperature. Turbine vanes generate frequently crack of thermal fatigue, which reduces its life. So resistance to thermal fatigue is one of the major criteria which should be considered when to select an alloy for a fluctuating temperature application. The microstructure significantly changes under the action of thermal stress generated by alternating temperature during thermal fatigue, which has deleterious effect on the mechanical property. However, the literature on the microstructure evolution of directionally solidified nickel base superalloy during thermal fatigue was almost not reported till now [3-4]. Therefore, the thermal fatigue microstructure changes of DZ951 alloy, which has the advantages of low density, low cost, high incipient melting temperature and better properties of thermal fatigue resistance and oxidation resistance [5], are systemically studied to determine the failure mechanism of thermal fatigue in this paper.

2. Experimental

The nominal composition of DZ951 alloy is (mass %): 0.05C, 9.0Cr, 5.0Co, 6.0Al, 2.2Nb, 3.0Mo, 3.0W, bal. Ni. The alloy first was melted in a VZM-25F type vacuum induction furnace. The directionally solidified specimens were made by high rate solidification (HRS) method in a ZGD2 type vacuum furnace with a temperature gradient of 60 – 80 °C/cm and a withdrawal rate of 6 mm/min. The thermal fatigue properties of as-cast alloy (AS alloy) and heat treated alloy (HT alloy) were investigated. The heat treated procedure was 1220 °C/4 h, AC (air cooling) + 1050 °C / 4 h, AC + 870 °C / 24 h, AC. Fig. 1 showed the specimen of thermal fatigue. Notched direction was perpendicular to the orientation of dendritic growth ([001] direction). Specimens were

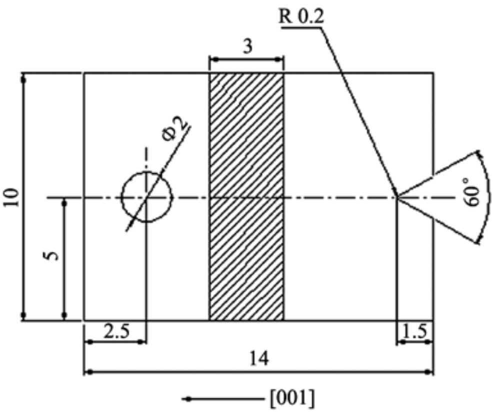

Fig. 1. Schematic diagram of thermal fatigue specimen (mm).

Corresponding author: XIA Pengcheng **E-mail:** xpc328@126.com

polished and examined before test. All of eligible specimens were required without crack near notch. Specimen was first heated to upper temperature. The heating time was 10 min. Then it was cooled to room temperature by water basin and the cooling time was 30 seconds. The upper temperature were respectively 1000, 1050 and 1100 °C. SEM and TEM were applied to examined the γ′ morphology and dislocation configuration after 150 cycles.

3. Results and discussion

3.1. γ′ denuded region

There was γ′ (Ni_3Al) denuded region in the two sides and tip of thermal fatigue crack after thermal fatigue experiment (indicated by arrow in Fig. 2 (a), (b)). According to Fig. 2 (b), the result of the line scanning demonstrated that the content of Al and Cr drops and the content of Ni increased in some regions (BC and DE in Fig. 2 (c)), which indicated that γ′ denuded phenomenon was related to oxidation. The elements near thermal fatigue crack are unstable. During thermal fatigue, it is easy for Al and Cr elements to combine with O in air and form oxide, which desquamate from the sides of crack under thermal stress in the crack surface due to the alternating temperature. It induces Al and Cr elements to continue oxidation so that the contents of Al and Cr gradually decrease in the sides and tip of thermal fatigue crack and the γ′ volume fraction reduces near crack with the drop of Al element. The longer the exposure time at high temperature, the more sufficient the oxidization course. On the other hand, the γ′ solution in the matrix increases for the drop of Cr element near crack, which further reduces γ′ volume fraction. There is γ′ denuded region. The γ′ denuded region in the crack tip is small for the short forming time of crack and uncompleted oxidation.

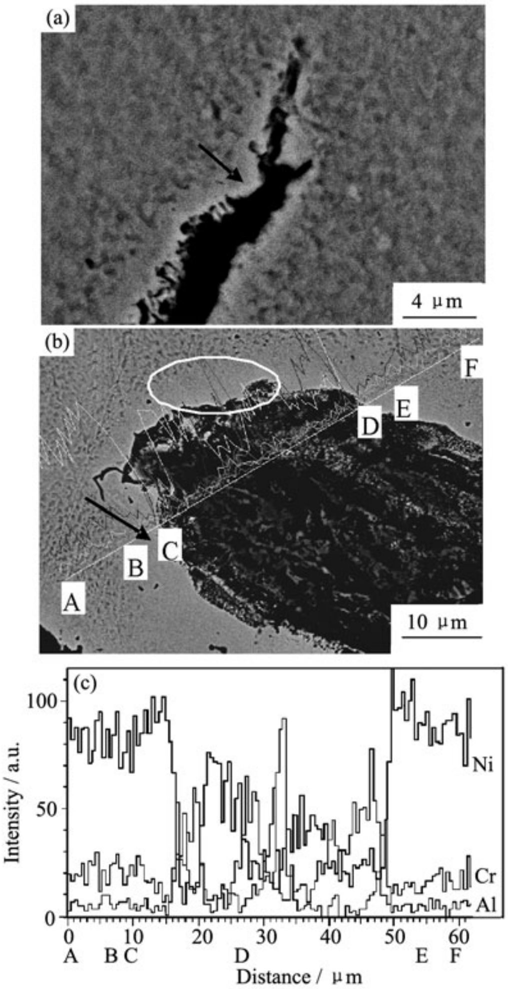

Fig. 2. γ′ denuded region in the course of thermal fatigue: (a) AS alloy at the upper temperature of 1050 °C (b) HT alloy at the upper temperature of 1100 °C (c) Line scanning corresponding to the γ′ denuded region in HT alloy.

3.2. γ′ rafting

The γ′ precipitate of AS alloy was irregular with even size of 400 μm (Fig. 3 (a)). After heat treatment γ′ phase became cubic in the size of about 300 μm (Fig.3 (b)).

γ′ phase coarsened and rafted during thermal fatigue. The rafting direction was perpendicular to the orientation of dendritic growth (Fig. 4). The rafting microstructure of AS alloy (Fig. 4 (a)) was more pronounced than that of HT alloy

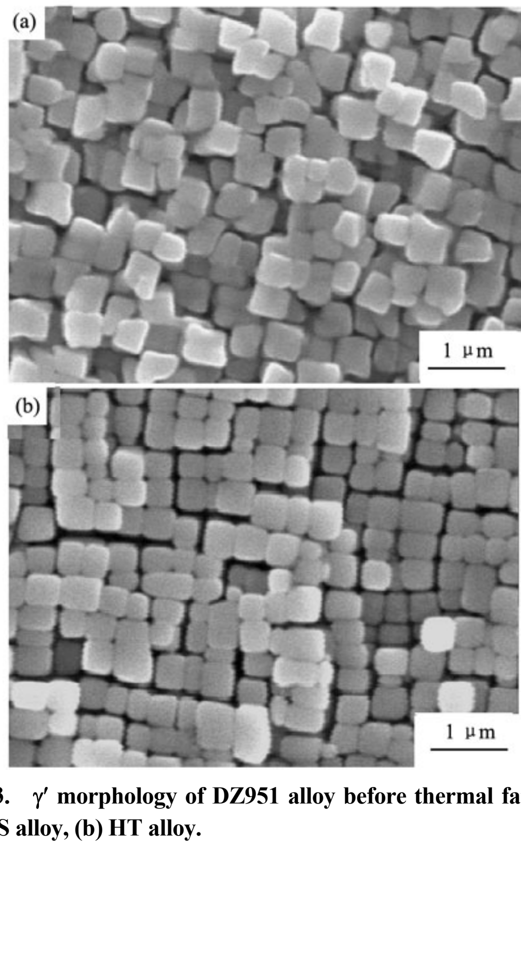

Fig. 3. γ′ morphology of DZ951 alloy before thermal fatigue: (a) AS alloy, (b) HT alloy.

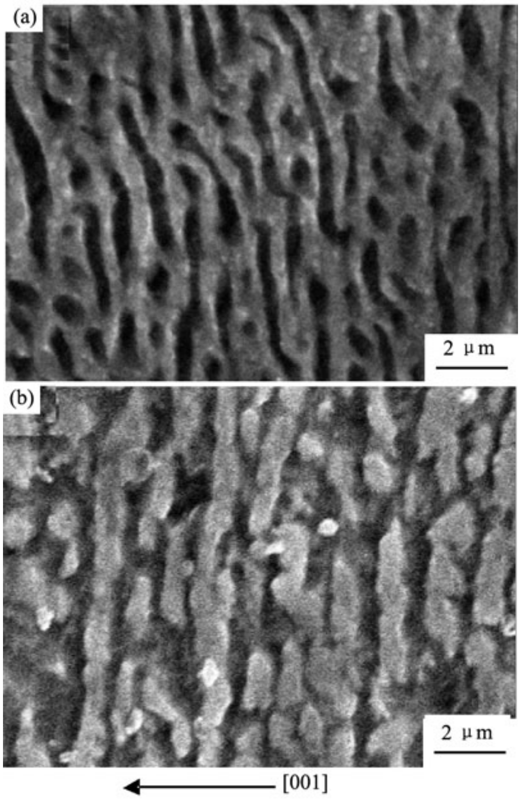

Fig. 4. γ′ rafting in the course of thermal fatigue: (a) AS alloy at 1100 °C, (b) HT alloy at 1050 °C.

(Fig. 4 (b)). The strength and stability of γ′ phase are significantly improved after heat treatment, which reduces the diffused rate of alloy element and makes HT alloy form imperfect rafting during thermal fatigue. γ′ rafting is related to the alternating temperature and thermal stress in the course of thermal fatigue. There is temperature gradient in the specimen during heating. The surface of specimen expands at high temperature but is restricted by the center so that compressing stress can be generated in the surface. The stress increases with the rise of temperature gradient. It is similar to the creep under compressing stress. The thermal stress gradually decreases when the temperature of all parts of the specimen near equal. It is the similar to the long-term exposure without stress. Furthermore, the surface of specimen shrinkages during water cooling to display tensile stress for the restriction of center of the specimen. It is similar to the creep under tensile stress. Cooling course has little effect on the γ′ rafting because of short cooling time. So γ′ particles intermittently raft under the compressing stress at elevated temperature during thermal fatigue. There are many literatures about γ′ rafting during creep and long-term exposure without stress [6-15]. The rafting direction is related with exterior stress and misfit between γ′ phase and matrix γ phase. The direction of γ′ rafting of the alloy with negative mismatch during compressing stress is parallel to the stress [9]. The rafting direction of superalloy during long-term exposure without stress is <100> [5]. The mismatches of AC and HT alloys are respectively –0.27% and –0.22%. The direction of γ′ rafting is perpendicular to the orientation of stress. It indicates that the effect of temperature on the direction of γ′ rafting is more than that of inner stress.

3.3. Dislocation configuration

No dislocation can be found between irregular γ′ precipitates of AC alloy (Fig. 5). It produces big stress in the notch in the action of alternating temperature during thermal fatigue. The plastic deformation of alloy happens under the stress more than the elastic strength. With the rise of cycles, cracks of thermal fatigue initiate and propagate to release thermal stress at high temperature. But dislocations occur in the bigger plastic deformation regions. Lots of dislocations presented in the regions of 2 mm away from thermal fatigue crack of AS alloy after 150 cycles at upper temperature of 1000 °C (Fig. 6 (a)). Some dislocations shearing γ′ phase and movement in the matrix could be seen in Fig. 6 (a). Many dislocations formed dislocation ranges with high density in the γ/γ′ interface near crack (Fig. 6 (b)). The amount of dislocations is related with the deformation degree. The larger the deformation the higher density of dislocations (Fig. 6 (b)). The density of dislocation decreases far away crack for small deformation (Fig. 6 (a)).

Fig. 7 showed the dislocation configuration of DZ951 alloy after 150 cycles at upper temperature of 1100°C. γ′ phase formed regular rafting at high temperature (Fig. 7 (a)). High density dislocations assembled around γ′ rafting (Fig. 7 (b)). It was difficult for dislocations to shear γ′ rafting. So some dislocations gradually started to climb because of heat activation at 1100 °C (Fig. 7 (b) and (c)). Many fine γ′ particles precipitated in the matrix (Fig. 7 (c)). This indicates that a few γ′ precipitates solutes into matrix at higher upper temperature. Fine and spherical γ′ particles precipitated during cooling (Fig. 7 (c)). Large amount of finer γ′ particles may be retard the movement of dislocations in the matrix, which is beneficial to thermal fatigue resistance.

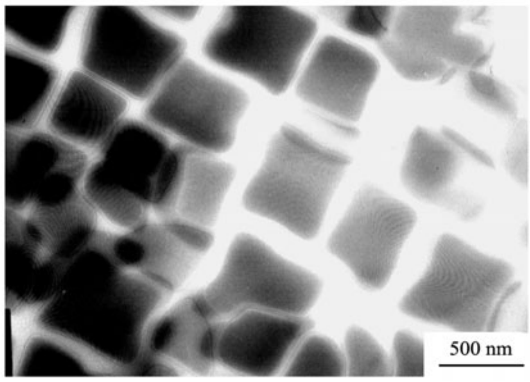

Fig. 5. TEM photo of AS alloy before thermal fatigue test.

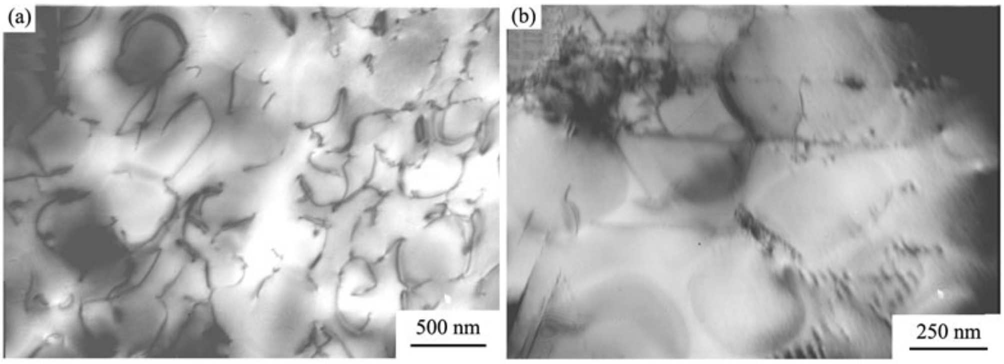

Fig. 6. Dislocation configuration of AS alloy after 150 cycles at 1000 °C single dislocation in the matrix (b) high density dislocations in the matrix.

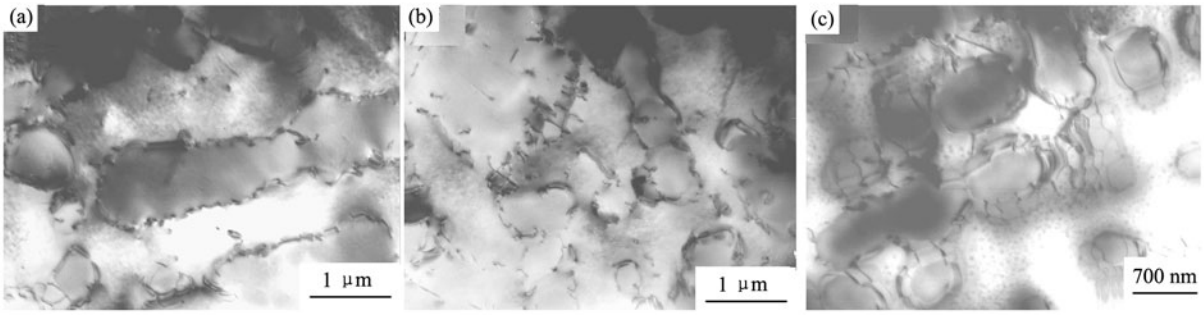

Fig. 7. Dislocation configuration of AS alloy after 150 cycles at 1100 °C: (a) γ′ rafting, (b) dislocations shearing γ′ phase, (c) fine and spherical γ′ precipitates in the matrix.

4. Conclusion

(1) There is γ′ denuded region in two sides and the tip of crack because of oxidation during thermal fatigue at DZ951 alloy. γ′ phase rafts perpendicularly to the direction of crystal growth in the actions of temperature and thermal stress.

(2) There are some dislocations owing to large plastic deformation during thermal fatigue experiment. High density dislocations are found near crack. Single dislocation moves in the matrix far away crack. A few dislocations shear γ′ phase because of relatively low strength at high temperature. Fine and spherical γ′ phase precipitates in the matrix for γ′ partly dissolution at 1100°C.

Acknowledgement

This work was financially supported by the National Basic Research Program (973 Program) of China (No. 2010CB631200 (2010CB631206)), the National Natural Science Foundation of China (NSFC) (No. 50931004).

References

[1] Sims C.T., Stoloff N.S., and Hagrel W.C., *Superalloy* II, John Wiley & Sons, New York, 1987: 3.

[2] Matthew J., and Donachie J., *Superalloy*, American Society for Metals, Ohio, 1984: 3.

[3] Woodford D.A., and Mowbray D.F., Effect of material characteristic and test variable on thermal fatigue of cast superalloys, *Mater. Sci. Eng.*, 1974, **16**: 5.

[4] Rarna V., and Sarma D.S., Influence of thermal fatigue on the microstructure of a Ni-base superalloy, *Scr. Metall. Mater.*, 1993, **29**: 467.

[5] Xia P.C., Yu J.J., Sun X.F., Guan H.R., and Hu Z.Q., Influence of thermal exposure on the microstructure and stress rupture property of DZ951 alloy, *J. Alloys Comp.*, 2007, **443**: 125.

[6] Murakumo T., Kobayashi T., Koizumi Y., and Harada H., Creep behaviour of Ni-base single-crystal superalloys with various γ′ volume fraction, *Acta Mater.*, 2004, **52**: 3737.

[7] Henderson P., Berglin1 L., and Jansson C., On rafting in a single crystal nickel-base superalloy at high and low temperature creep, *Scr. Mater.*, 1999, **40**: 229.

[8] Sarosi P.M., Srinivasan R., Eggeler G.F., Nathal M.V., and Mills M.J., Observations of a <010> dislocations during the high-temperature creep of Ni-based superalloy single crystals deformed along the [001] orientation, *Acta Mater.*, 2007, **55**: 2509.

[9] Fredholm A., and Strudel J.L., On the creep resistance of some nickel base single crystals, [in] *Superalloys 1984*, Warrendale, 1984: 211.

[10] Acharya M.V., and Fuchs G.E., The effect of long-term thermal exposures on the microstructure and properties of CMSX-10 single crystal Ni-base superalloys, *Mater. Sci. Eng.*, 2004, **A381**: 143.

[11] Epishin A., Link T., Klingelh H., Fedelich B., Brückner U., and Portella P.D., New technique for characterization of microstructural degradation under creep: Application to the nickel-base superalloy CMSX-4, *Mater. Sci. Eng. A*, 2009, **510-511**: 262.

[12] Cheng K.Y., Jo C.Y., Kim D.H., Jin T., and Hu Z.Q., Influence of local chemical segregation on the γ′ directional coarsening behavior in single crystal superalloy CMSX-4, *Mater. Charact.*, 2009, **60**: 210.

[13] Xia P.C., Yu J.J., Sun X.F., Guan H.R., amd Hu Z.Q., Influence of thermal exposure on γ′ precipitation and tensile properties of DZ951 alloy, *Mater. Charact.*, 2007, **58**: 645.

[14] Fabienne T., Eric A., Dominique P., and Bernard V., Rafting microstructure during creep of the MC2 nickel-based superalloy at very high temperature, *Mater. Sci. Eng. A*, 2009, **510-511**: 244.

[15] Yu J.J., Lian Z.W., Chu Z.K., Sun X.F., Guan H.R., and Hu Z.Q., Properties and microstructures of M951 alloy afterlong-term exposure, *Mater. Sci. Eng. A*, 2010, **527**: 1896.