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Effect of Shot Peening on the Surface Integrity and Notched Fatigue Properties of a Single-crystal Superalloy at Elevated Temperature

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Abstract: The surface integrity and notched fatigue property of a shot-peened single crystal superalloy were investigated. The surface texture of the peened material was determined using a white light interferometer, while the microstructural characterization was performed using a scanning electron microscopy, high resolution transmission electron microscopy from the plane and cross-section view directions and electron backscatter diffraction. The gains, expressed in term of high temperature fatigue limit, were established by rotating-bending fatigue tests and discussed in relation to the microstructure evolution in the shot peening states. Results show that the beneficial effect provided by shot peening is more than 14.8% against the as-machined in a temperature range from 760 °C to 850 °C because of the high-density tangled dislocation, cold work and disorientation. Furthermore, the stress concentration coefficient is reduced by the shot peening procedure while the average profile height R_a increases according to the calculation of the surface stress concentration.

Key words: high cycle fatigue; shot peening; single crystal superalloy; disorientation

The performance of gas turbines partly relies on the temperature capability of the turbine blades. Conventional polycrystalline-material blades will creep along the grain boundaries at elevated temperatures of 1350 K. Thus, single crystal superalloys (SC) are widely applied in the hot sections of aero-engines (such as high-pressure turbine blades)^[1,2] because of their excellent creep resistance. Turbine blades are exposed to thermal and mechanical loading, particularly in high cycle alternative stress as a result of high frequency vibrations in flight. For this reason, fatigue failure, especially in the stress-concentration region (e.g. rabbet and gas film hole), is one of the major problems in the service of nickel-base SC^[3]. Hence, it is of great significance to focus on the fatigue life extension of SC.

To improve the fatigue strength, the crack initiation and propagation at the surface should be restricted by surface

mechanical treatments. The mechanical surface treatment method such as shot peening, cold expansion, and laser shock peening, improves the fatigue performance of metal parts because of the beneficial compressive residual stress and microstructure refinement near the surface. Shot peening is a low-cost and effective method to alleviate the fatigue performance of mechanical parts. Many researches focus on the anti-fatigue effects of shot peening on the steel^[4], titanium alloy^[5], and aluminum alloy ^[6]. However, many previous studies based on shot peening on SC have investigated the effect of recrystallization as a result of surface deformation and high temperature aging rather than fatigue. Jin et al.^[7] discussed the effect of a recrystallization layer by shot peening on creep rupture properties and found that the creep rupture in SC decreases with an increase of the recrystallized depth. Zhang et al.^[8] experimentally investigated the recrystallization

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of shot-peened SC influenced by the dissolution of primary γ' precipitates.

Recrystallization arises at the appropriate annealing temperature, annealing time and deformation degree with the release of surface deformation energy. Obviously, recrystallization would not occur in a wide range of relatively low temperatures, while mechanical surface treatment may efficiently promote the fatigue strength in the temperature range. Typically, Gao et al.^[9] and Wang et al.^[10] investigated the effect of shot peening on DD6 SC, and found that shot peening could be beneficial for extending the fatigue life at 650 °C. Nevertheless, the strengthening effect of shot peening could relax the fatigue process, especially at elevated temperatures. Compressive residual stress due to shot peening is the main mechanism to extend the fatigue life^[11]; however, peening residual stress is unstable during fatigue, especially at elevated temperatures^[12]. Dalaei et al.^[13] studied the stability of residual stress induced by shot peening and its influence on the fatigue lifetime of normalized steel and found that the stability of residual stress was determined by the plastic strain amplitude and number of cycles during the fatigue loading. Nalla et al.^[14] proposed that the extent of the relationship could be defined as a function of the exposure temperature; likewise, a near surface "case" would progressively degrade when the ratio of exposure at temperature T and melting point $T_{\rm m}$ was more than 0.4. Since, when raising the temperature to 760~850 °C, the ratio for SC is more than 0.4, the peening residual stress may relax to a relatively low level. Furthermore, the means to measure the residual stress of an anisotropic alloy is still not mature; therefore, the compressive residual stress after shot peening is not discussed in this paper. On the other hand, the effect of both surface texture and deformation micro-structure on the fatigue performance of peened SC at elevated temperature is sporadically discussed.

There has been little research on the effect of peening on SC. Therefore, the aim of this study is to systematically demonstrate the relationship between surface integrity and the notched fatigue property of SC by comparing the results of as-machined and shot-peened SC at elevated temperatures.

1 Experiment

The chemical composition of the SC is listed in Table 1. The SC specimens, $\Phi 15 \text{ mm} \times 160 \text{ mm}$, with [001] orientation were cast by a crystal selection method in the directionally solidified furnace with high temperature gradient. The cylinder axis was parallel to [001] direction. The angle deviation was less than 15°. The mechanical property of the SC smooth specimens is shown in Table 2.

The solution and aging heat treatment procedure is shown as follows: 1310 °C/2 h/AC+1120 °C/4 h/AC+900 °C/4 h/AC. The specimens for observing the microstructure were etched with CuSO₄+HCl+H₂O. The resulting microstructure consisted of matrix phase γ and strengthening phase γ' , shown in Fig.1.

 Table 1
 Chemical composition of SC in the present

 work^[15] (wt%)

		WOLK	(""	/0)				
Cr	Со	W	Al	Та	Мо	Re	Hf	Ni
7.0	7.5	5.0	6.2	6.5	1.5	3	0.15	Bal.

Table	2 Mechanical	Mechanical properties of the SC smooth specimens					
T/°C	$\sigma_{ m b}/{ m MPa}$	$\sigma_{0.2}/\mathrm{MPa}$	$\delta_5/\%$	ψ /%			
760	1109	880	9.6	13.5			
850	1072	1044	22.12	22.18			

The fatigue specimens shown as Fig.2 are illustrated as a notch-bar with nominal stress concentration coefficient K_t =1.7. All the specimens were manufactured by wire-electrode cutting, turning and grinding step by step. The specimens were sectioned into coupons by as-machined (AM) and shot-peened (SP) to a target Almen intensity of 0.25~0.35 A using AZB600 ceramic shot (AMS 2431/7) composed of zirconia and silicon.

The surface texture was tested using a Shift Phase MicroXAM white light interferometer, which measured the optical path difference as the function of surface texture, and the 3D profile was shown. A great deal of textual data was obtained from the test of the white light interferometer, including the average roughness (R_a), root mean square roughness (R_q), mean spacing of asperities at the level of the central line (R_{sm}), Kurtosis value (R_{ku}), maximum peak to valley (R_i) and average peak to valley (R_z)^[16].

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Fig.1 Microstructure of the SC (the etched strengthening phase γ' is shown as the square of the cases, and matrix phase γ is shown as the edge of the cases)



Fig.2 Drawing of fatigue specimens (a semicircle notch is designed with the radius of 0.75 mm, whereas the nominal stress concentration coefficient is 1.7)

To discover the microstructure of peened SC, the crosssection of specimens, sectioned parallel to the peening direction, was ground using water-lubricated silicon carbide to $R_a=0.2 \mu m$, and then electro-polished to achieve a planar finish. The grain size and quantitative crystallographic data in the area of 200 µm×200 µm were acquired using a scan electron microscopy JSM JEOL-6010 with an electron backscattering diffraction (EBSD) device branded Apollo 300, with a 30 kV accelerating voltage and 10 nA probe current. During the test by SEM, the phase γ' phanerocrystalline was corroded after polishing. The post-processing of the EBSD data was handled using Oxford Instruments HKL Channel 5 software. The two scan paths of disorientation of the shot peening state are shown in Fig.3. Besides, the JEOL-2010 high resolution transmission electron microscope (HRTEM) was used to observe the microstructures of the "case". The two direction views (cross-section and plane) were used. The cross-section specimen preparation method by HRTEM is the same as the document ^[17]. The plane view of the surface "case" of TEM foils, cut parallel to the (001) plane, was obtained by polishing the sample mechanically on the untreated side until it was about 30 µm thick, followed by ion milling using a Gatan PIPS with a small incident angle. Additionally, the hardness profile was measured on the carefully polished cross-section, using an FM-700 microhardness tester as a function of the depth when peened and after fatigue process.

Rotating-bending 'notch-bar' stress/life (S/N) fatigue tests with stress ratio -1 were performed under load-control using a fatigue testing machine operating at a frequency of 83.3Hz (sine wave). Both the AM and SP state specimens were measured at 760 and 850 °C. The temperature of the furnace was continuously monitored by a k-type thermo-couple placed in close proximity to the specimens. The fatigue fractures were observed using a Quanta-600 scan microscopy.

2 Results and Discussion

The effect of shot peening on fatigue is composed of three factors: texture modification, surface microstructure enhancement



Fig.3 Two paths of analyzed disorientation (path 1 is along the depth direction, and path 2 is in the target material as a contrast)

and compressive residual stress. The residual stress was not discussed in this study. Other factors, fatigue property and fractographic observations, were investigated in the subsequent sections.

2.1 Surface texture

Fig.4 shows the AM and SP surface textures, indicating that shot peening provokes the deformation of the SC surface. Compared to the AM surface texture with parallel tool marks due to grinding, a few micrometric dents, the diameter of which varied approximately from 150 μ m to 190 μ m, were observed on the SP surface. The parameters (ISO 13565-2) for surface roughness are given in Table 3. The shot peening led to an increase of R_a , R_q , R_{sm} , R_t and R_z ; likewise, the R_{ku} declined after the shot peening.

The geometrical stress concentration factor K_t plays an important role in the fatigue performance, calculated by Eq.(1) or Eq.(2), depending on the ratio of R_z/R_{sm} by Li et al^[17], Clausen and Stangenberg^[18] and Rodopoulos et al^[19,20].

$$K_{\rm t} = 1 + 4(\frac{R_{\rm z}}{R_{\rm sm}})^{1.3}$$
 when $\frac{R_{\rm z}}{R_{\rm sm}} < 0.15$ (1)

$$K_{\rm t} = 1 + 2.1(\frac{R_{\rm z}}{R_{\rm sm}})$$
 when $\frac{R_{\rm z}}{R_{\rm sm}} \le 0.3$ (2)

In this case, the mean stress concentration is 1.7, instead of the constant "1". The AM and SP ratios of R_z/R_{sm} are 0.26 and 0.22, respectively; therefore, the calculated stress concen-



Fig.4 Surface texture of the specimens AM (a) and SP (b)

Table 3 Parameters for the surface roughness of the AM and SP condition

Condition	<i>R</i> _a /nm	R _q /nm	$R_{\rm ku}$	R _{sm} /nm	R _t /nm	R _z /nm
AM	432	543	3.91	15730	4429	4090
SP	1236	1589	3.61	84518	19098	18432

trations are 2.24 in AM as opposed to 2.162 in SP. This reveals that the stress concentration decreases by 3.5% because of shot peening, despite the increase of the average profile height R_a reported in our previous study^[21]. This may be attributed to dents caused by shot peening, sharing the applied load to reduce the stress amplitude. In summary, a shot peening texture optimizes the AM surface, and is beneficial for improving the fatigue property of SC.

2.2 Surface microstructure

The AM and SP near-surface SEM microstructures are shown in Fig.5. Compared with Fig.1, the single-crystal alloy's microstructure has significantly changed by shot peening. Excluding the matrix, two modes of microstructure were investigated on the peened "case". On the outer layer, which is $3\sim5$ µm indepth , the cubic γ phase and γ' phase could not be observed clearly, indicating that it was a severe deformation layer and the high-speed shot impact destroyed the cubic structure. Moreover in the inner layer of the depth $5\sim150$ µm, "line clusters" from two directions were investigated, cross and pierced through the cubic γ phase and γ' phase. The cubic lattice deformed through which "line clusters" pass, while the distal micro-structure kept cubic.

Fig.6 is the peened SC's cross-section transmission electron microscope microstructure. The incidence direction of electron beam is [110]. Using a larger magnification of TEM with the contrast of Fig.5 and Fig.6, the first, the "line cluster" formed by the parallel "fine lines" observed by the transmission electron microscope; the second, the length direction of "fine lines" was along the [111] direction. The [111] direction is the main direction of the face-centered cubic metal. Furthermore, after HRTEM test as Fig6c, 6d, it is proved that a fine line was a deformation twin grain.

From the plane view direction, the high-resolution image of the AM microstructure, diffraction pattern of the AM microstructure, high-resolution image of the SP microstructure and diffraction pattern of the SP microstructure are presented in Fig.7a~7d, respectively. The microstructure observation was performed using the same incidence direction of electron beam [100] in both the matrix phase γ and enhanced phase γ' to compare the dislocation configurations and densities. Above all, no recrystallization was observed.

The starting microstructure of the supplied SC is shown in Fig.7a. The microstructure consists of a large volume fraction of strengthening phase γ' with an average width of 500 nm. The matrix phase γ is visible between the strengthening phase, and a good coherent relationship between the two phases can be also observed in Fig.7b. In the "case", the dislocation arrangement consisted of diffuse tangled and debris-like structures, whereas no cell formation was observed. The shot peening led to a significant increase in the near-surface dislocation density in both phase γ and phase γ' . More dislocation can be seen in phase γ , compared with phase γ' . This may be attributed to there being more slip planes of face-centered cubic (space group No.Fm-3m^[22]) in phase γ than in phase γ' .

The EBSD images are shown in Fig.8, whereas 95% confidence bars are based on 250 000 data points. In the SP state, the EBSD data reveal the disorientation of the "case" in Fig.8a. The disorientation angle compared to the orientation [001] of surface crystals as a function with the depth is shown in Fig.8b. As the same of cross-section SEM results, a sandwich structure was observed in the "case" of the peened



Fig.5 Overall view of peened microstructure at 2000× (a), the severe deformation layer at 20000× (b), the inner layer at 5000× (c), and the amplified view of inner layer at 20000× (d)



Fig.6 Microstructure at 40000× magnification (a) and SAED pattern at 150× magnification of SP (b) using a high resolution transmission electron microscopy from the cross-section view direction; HRTEM microstructure at 500 000× magnification (c) and HRTEM microstructure at 500000× magnification processed by Mapvue AE V2.24 (d)



Fig. 7 Microstructure at 10000× magnification and SAED pattern at 150× magnification of AM (a, b) and the microstructure and SAED pattern of SP (c, d) using a high resolution transmission electron microscopy from the plane view direction

material. From the depth of 0 μ m to 5 μ m, the disorientation increased from 0° to 1.5°. This depth was basically consistent with the severe plastic deformation layer observed by cross-section SEM. The second layer contained a disorientation of

 1.5° with a depth of 45 µm, whereas peening yielded the plastic deformation of this layer with the fabrication of disorientation. Moreover, 8° of accumulated disorientation was observed in the third layer in a depth range of 45 µm to



Fig. 8 Cross-section EBSD map of peened single crystal (a); the top of the map corresponds to the shot-peened edge, disorientation as a function of depth (b); disorientation along path 2 in the matrix material (c); low angle boundaries from 0.5° to 2° based on the EBSD data (d)

150 μ m, which is consistent with "the inner layer" viewed by SEM. It is the shot peening influence layer, with relative little deformation. Instead, the max disorientation angle was compared with peened "case", less than 0.7° in the matrix material SC, as shown in Fig.8c. The results prove that peening constructs the layer structure. Furthermore, as can be observed in Fig.8d, plenty of low angle boundaries at the range from 0.5° to 2° were shown close to the surface, while the depth, 0~45 μ m, was consistent with the first layer. We anticipate that the plastic strain produced during shot peening is also formed by the dislocation glide and the tiny rotations of crystals.

Slips play an important role in the mechanical process of SC^[23] at an elevated temperature. Matan et al.^[24] suggested that the extent of the creep strongly depends on a small disorientation away from the <001>/<011> symmetry boundary at 750 °C, whereas the effect of disorientation is weaker at 950 °C. However, disorientation was observed on the surface in this study. The existence of disorientation increases the stacking fault energy in the peened "case" and hinders the slip in the "case" during the fatigue process. On the other hand, the microstructure of the peened "case" is relatively stable in elevated temperatures, compared with the residual stress ^[25]. Eleiche et al. ^[26] suggested that the rotation of the surface crystal was the main reason for the strengthening effect of shot peening at elevated temperatures and it is believed that the peening disorientation and layer structure would extend the fatigue life at such elevated temperatures.



Fig.9 Microhardness profile of shot-peened SC

The micro-hardness represents the resistance of deformation by applied load. Fig.9 is the micro-hardness profile of peened SC. Micro-hardness value is the highest on the surface, decreases with the depth and then back to the same level as the matrix at about the depth 100 μ m. This variation trend is basically the same as the microstructure deformation. Thus, it is believed that the plastic deformation induced by shot peening causes the increase of surface micro-hardness and the deformation resistance improvement of applied load. The cold work could lead to the promotion of fatigue property.

2.3 Notch fatigue property

Fig.10 shows the S-N data for the peened and as-machined samples at 760 and 850°C, respectively. It can be seen from



Fig.10 Maximum stress (σ_{max}) vs. number of cycles to failure (N_f) in the state of peening and as-machined at 760 °C (a) and 850 °C (b) with notched fatigue specimens (K_i =1.7)

this figure that the shot peened specimens illustrate an increase of fatigue life compared to the as-machined specimens across the whole range of stress amplitudes. For example, in the low stress regime of 380 MPa, the notch

fatigue life of the SP state was increased by the appropriate order of magnitude to the AM state specimens at 760 and 850 °C. Instead, the average increase in the fatigue life of SP was diminished under higher stress. In terms of the stress of 500 MPa, the average notch fatigue life of the SP state was extended by about 680% relative to the AM state specimens at both temperatures. The 10^7 -cycles-conditional fatigue limit of the AM specimens at 760 °C was 330 MPa, while it was 379 MPa of the peened specimens, representing a rise of 14.8%. Similarly, there was also an increase in the endurance limit at 850 °C, from around 320 MPa to 385 MPa, an increase of 20.3% in the available applied stress amplitude.

Overall it can be concluded that the peening procedure provides a beneficial improvement in the fatigue life of a single crystal superalloy, with the optimal texture hindering the initiation of cracks. Disorientation in a single crystal superalloy, cold work and high density dislocation structure provide positive resistance to the propagation of fatigue cracks.

2.4 Fractographic observations and microhardness after fatigue

In order to explain the influence of shot peening on fatigue failure, fatigue failure was observed using a scanning electron microscopy. The crack was propagated along $\{111\}$ octahedral slip planes at 850 °C; on the contrary, it expanded perpendicular to the loading axis of specimen at 760 °C. This significant difference suggests that main slip system may be transformed when the temperature is higher than 760 °C. At both temperatures the fatigue cracks of the peened specimens nucleated at the peening dents. Then, the crack propagated with the formation of a rough platform at a maximum stress of



Fig.11 Fatigue fractures of peened specimens (left), and fatigue sources (white arrows of left ones) are shown as right ones: (a, b) 760 °C, σ_{max} =500 MPa, $N_{\rm f}$ =5.67×10⁵ cycle; (c, d): 850 °C, σ_{max} =500 MPa, $N_{\rm f}$ =7.74×10⁵ cycle

500 MPa. The depth of the rough platform, 130 μ m, is in agreement with the shot peening-influenced layer. In contrast, at the same stress, the AM fatigue source was relatively smooth as shown in Fig.12. It is believed that disorientation and a high-density structure hinder the propagation of fatigue crack and lead to a rough source of fatigue, whereas the propagation path becomes tortuous.

The micro-hardness profile of the sample after peening and fatigue process was investigated as Fig.13. The hardness increase by peening deformation still existed. However compared with Fig.9, the value of hardness and the depth of peening layer decrease. During the high temperature fatigue process and under the applied load, shot peening enhanced layer relax by the annihilation of contrary sign dislocation, the



Fig.12 Fatigue fractures of as-machined specimens: (a) 760 °C, σ_{max} =500 MPa, $N_{\rm f}$ =1.37×10⁵ cycle; (b) 850 °C, σ_{max} =500 MPa, $N_{\rm f}$ =1.25×10⁵ cycle



Fig.13 Microhardness profile of shot-peened SC before and after fatigue process

movement and piling up of dislocation at the surface or sub-grain boundary, and the high temperature facilitated the process. In any case, after the fatigue process, the microhardness results show that the cold work effect still exists but has weakened.

3 Conclusions

1) The peened single crystal superalloy shows a significantly higher surface roughness than the as-machined. Meanwhile, shot peening decreases the geometrical stress concentration factor $K_{\rm t}$, indicating that peening is beneficial to the surface texture for extending the fatigue life.

2) In SEM, the outer layer is a severe deformation layer in which the cubic γ phase and γ' phase can not be observed. Moreover, a "line cluster" structure of about 150 µm in the inner layer is investigated, and with a larger magnification of TEM, the "line cluster" is formed by the parallel twin grain.

3) A sandwich structure is observed by EBSD: the first layer caused by severe deformation, is about 5 μ m depth with the consistence of outer layer of cross-section SEM; the second layer, containing 1.5° disorientation, is appropriately 45 μ m in depth; meanwhile, the next layer has an accumulated disorientation of about 8° located at the depth range of 45~150 μ m. Indeed, there are more dislocations in the phase γ than in γ' , which form diffuse tangled and debris-like structures. Micro-hardness value is the highest on the surface, decreases with the depth and then back to the same level as the matrix at the depth of ~100 μ m. This variation trend is basically the same as the microstructure deformation.

4) Shot peening makes a significant improvement in the fatigue life at temperatures of 760 and 850 °C, with an increase in the fatigue life of more than 6 times, depending on the applied stress amplitude, and 14.8% and 20.3% increase in the endurance limit of single crystal superalloy at temperatures of 760 and 850 °C. Additionally, a crack is propagated along {111} octahedral slip planes at 850 °C, while it grows perpendicular to the loading axis at 760 °C. After both temperatures, fatigue cracks nucleate at the peening dents and propagate with the formation of a rough platform, which is attributed to the disorientation, cold work and high-density dislocation structure by peening. In any case compared with the state after peening, after the fatigue process, the micro-hardness results show that the cold work effect still exists but has weakened.

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喷丸对一种单晶合金表面完整性和高温缺口疲劳性能的影响

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摘 要:研究了喷丸后单晶合金的表面完整性和高温缺口疲劳性能。喷丸后表面形貌采用白光干涉仪表征,扫描电镜,2个方向观察(平 行于受喷表面和截面方向)高分辨透射电镜和电子背散射衍射分析,疲劳性能采用旋转弯曲疲劳模式表征。采用旋转弯曲疲劳极限表征 了强化增益作用,并与喷丸显微组织建立联系。结果表明,在760℃到850℃区间,喷丸强化较原始加工状态的疲劳极限增益达到14.8%, 主要原因是高密度的缠结位错、加工硬化和晶粒错配。此外,通过表面应力集中系数计算表明,即使在平均粗糙度 *R*a 升高的基础上, 喷丸强化后表面应力集中系数也降低。

关键词: 高周疲劳; 喷丸; 单晶合金; 晶粒错配

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