PAPER

Effect of stress-relief annealing on isothermal fatigue life of a new hot stamping die steel 4Cr2Mo2V

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Keywords: isothermal fatigue, hot stamping, stress-relief annealing, hot work die steel

Abstract

For 4Cr2Mo2V hot stamping die steel, the isothermal fatigue test was suspended at different cycles, and the stress-relief annealing was carried out in situ. After annealing, the fatigue tests were continued. The stress-relief annealing was carried out at 30%, 50% and 70% of the cycle life when the total strain amplitude was 0.5%, and the fatigue life was increased by 30.27%, 23.84% and 10.24% respectively. On the basis of Basquin-Coffin-Manson life prediction model, by adding the influence of stress-relief annealing on life, obtained the stress-relief annealing life prediction model. The dislocation density, microstructure and pole figure were analyzed by TEM and XRD to explore the mechanism of stress-relief annealing on prolonging fatigue life. After stress-relief annealing, the full width at half maximum (FWHM) decreased, and the dislocations originally piled up at the grain boundary moved into the grain, delayed the time of dislocation cell or wall formation. At the same time, it could also eliminate the trend of consistent orientation of most grains in the fatigue process. This is the main reason why stress-relief annealing prolongs fatigue life.

1. Introduction

The service life of hot stamping die is closely related to the in-depth maintenance, but in the actual production process, the in-depth maintenance of die is often not paid attention to, and more die maintenance is shallow. For example, with the application of various surface treatment technologies (polishing, nitriding, oxidation, PVD, etc), the role of these surface treatment is more to delay the degree of friction and wear of the die, especially the edge. Although the friction and wear of the edge is one of the main failure forms affecting the hot stamping production process [1, 2]. But the greater harm is the mold cracking and water leakage. the place where the die cracks is treated by repair welding (most need annealing) [3, 4], it often takes more than 3 days, which seriously affects production. The deep maintenance of the die requires stress-relief annealing after a certain number of stamping dies. The annealed die still needs stress-relief annealing again after it continues to be used for a period of time, so as to maximize the service life of the die. When to carry out stress-relief annealing is often based on experience, and there is no accurate quantitative theoretical research that can guide the practice.

The classical Coffin Manson formula [5, 6] is usually used for the calculation of fatigue life:

\[ \frac{\Delta e_{pm}}{2} = \varepsilon'_f (2N)^{\nu} \]  

with total plastic strain amplitude at half-life \( \Delta e_{pm} \), fatigue ductility \( \varepsilon'_f \), fatigue ductility exponents \( c \). On the basis of this classical formula, most scholars have modified the formula to adapt to more fatigue environments, such as Basquin-Coffin-Manson model widely used in thermomechanical fatigue [7, 8]:

\[ \frac{\Delta e_m}{2} = \frac{\sigma'_f}{E} (2N)^{\nu'} + \varepsilon'_f (2N)^{c'} \]  

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with total strain amplitude at half-life $\Delta \epsilon_{\text{sh}}$, elastic modulus $E$, and $\sigma'$ and $b'$ are material constants. Used this method to predict the low cycle fatigue life of martensitic forging die steel at high temperature, and obtained good results \[9\].

It is known that stress-relief annealing eliminates the internal residual stress of the material. Due to the low temperature, it will not change the mechanical properties of the material, but can significantly affect the fatigue life. Because welding produces a large amount of residual stress. Therefore, this research is mostly seen in the fatigue test after welding \[10, 11\]. For example, Kai Xu et al \[12\] studied the fatigue properties of X80 steel after welding through low-temperature stress-relief annealing, it can not only eliminate the residual stress, but also change the microstructure inside the grain, so that the high-energy dislocations accumulated at the grain boundary move into the grain \[13\]. These are studied by stress-relief annealing before fatigue test, and the above life model can still be used for fatigue life prediction. The tests are suspended during the tests, full stress-relief annealing was carried out, and then the fatigue tests were continued. There are few studies on the effect of midway stress-relief annealing on fatigue life. Nevertheless, this process of stress-relief annealing is widely used in hot stamping production (based on production experience). Therefore, this paper will study stress-relief annealing in different life stages, quantitatively study the effect of stress-relief on fatigue life, and deeply explore its mechanism.

2. Experimental procedure

2.1. Material
The test material is 4Cr2Mo2V hot work die steel widely used in hot stamping \[14, 15\]. The chemical composition of the 4Cr2Mo2V is 0.46 C, 0.42 Si, 0.66 Mn, 2.56 Cr, 2.35 Mo, and 0.82 V (in wt. % unit). The billet was remelted by Electroslag Remelting (ESR), and all samples were cut from the section of high-quality billet module. Samples were quenched at 1030 °C and tempered at 570 °C, two times of tempering, each time for 2 h. The final hardness of all samples is 54 ± 1HRC. The microstructure of the material is tempered martensite and a large number of carbides are dispersed. The microstructure of the material is shown in figure 1.

2.2. Fatigue tests
MTS 809 axial/torsional test system was adopted for the test equipment. The resistance furnace was used to heat samples. Temperature was controlled within ±2 °C during the period of the tests, and all test temperatures were 300 °C. The test equipment, sample loading diagram and sample size are shown in figure 2. Isothermal fatigue tests were carried out under fully reversed tension-compression loading conditions with different cycles. Total strain amplitudes were ±0.5%. The ratio of minimum strain to maximum strain ($R$) was $-1$, and the frequency was 0.25 Hz. The loading method was triangular wave. The video extensometer was used for strain detection, and the micro deformation distance was analyzed according to the image, so as to accurately control the total strain. After the tests were suspended, the in situ stress-relief annealing was carried out, and then the tests were continued. The connecting rod was water-cooled. In order to eliminate the influence of thermal stress, kept the sample at 300 °C for 20 min, and then tightened the upper grip for test, which was equivalent to clearing the thermal strain, and the applied mechanical strain was the total strain. The test conditions are shown in table 1. SBA and SAA were used to represent the samples before and after stress-relief annealing. Stress-relief annealing was suspended at 30%, 50%, 70% of the total life, respectively.
2.3. Microscopy
To investigate the variation of dislocation structure and the full width at half maximum (FWHM) as a function of the number of cycles or stress-relief annealing. The samples for transmission electron microscopy (TEM), X-ray diffractometer (XRD) observations were taken parallel to the loading axis from the gage section in the specimens fatigued and stopped at different cycles. Dislocation structure was examined using JEM-2010F TEM operating at 200KV. FWHM and pole figure were investigated using Rigaku SmartLab 9KW x-ray diffractometer, scanning speed was 5° min$^{-1}$. Both SBA and SAA samples were tested by XRD.

3. Results

3.1. Cyclic peak stress response behavior after stress-relief annealing
Study the effect of stress-relief annealing on fatigue life, and compare the changes of cyclic peak stress curve before and after stress-relief annealing. Select SBA-2/SAA-2, that is, the samples suspended when the cycles were 50% of the fatigue life, for detailed analysis. As can be seen from figure 3(a), the cyclic peak stress curves of the samples before and after stress-relief annealing are similar, but there are still some differences. The first is the initial peak stress. Compared with the samples without stress-relief annealing, the samples after stress-relief annealing are reduced, but the reduction range is not obvious; The second is the change of the second stage. The number of cycles and response peak stress of the sample entering the second stage are different. As shown in the small figure of figure 3(a), the initial response stress of the sample entering the second stage during the cycle is reduced by 15.64% compared with the SBA sample, which is also one of the reasons for the significant increase of the fatigue life of the SAA sample. Because of the well-known common sense, under the same strain, the smaller the response stress is, the better the toughness is, which is conducive to the increase of service life. However, it is obvious that stress-relief annealing does not change the hardness of the material and does not affect the service performance of the material. This phenomenon is related to the reduction of dislocation density by stress-relief annealing [16, 17]. In addition, it can be seen from figure 3(b), The number of cycles entering the second stage is different. Because the initial response stress of SAA sample entering the second stage is low, it enters the second stage through more cycles. The number of cycles of SAA sample entering the second stage is 28.43% longer than that of SBB sample; there is another obvious change in the second stage, and the softening rate is different. Linear fitting is carried out for the second stage. To a certain extent, the slope can

![Figure 2. Fatigue test equipment and sample loading diagram.](image-url)
reflect the softening rate. As shown in figure 3(c), the slope of SAA sample is low and the softening rate is gentle, while the softening rate of SBA sample is significantly higher than that of SAA sample, more than 4.5 times. The softening rate is related to the microstructure evolution, dislocation density and arrangement during cycling \[17, 18\], and it is obvious that the gentle softening rate greatly prolongs the service life of the sample.

3.2. Effect of stress-relief annealing on fatigue life

Through three fatigue tests, the average isothermal fatigue life of 0.5% strain amplitude is 29919 cycles. Suspended the test at 30%, 50% and 70% of the average life respectively, then loosened the upper grip and conducted in situ stress-relief annealing for the sample, and continued the isothermal fatigue tests after annealing. The test results are shown in figure 4. SBA and SAA samples are red and green respectively. After stress-relief annealing, the life of all samples is extended to varying degree. The total life is shown in blue. Compared with the average fatigue life under this strain, the total life increase rates after three stress-relief annealing are 30.27%, 23.84% and 10.24%, respectively.
4. Discussion

4.1. Establishment of stress-relief annealing fatigue life model

Figure 4 shows that the fatigue life can be significantly increased by stress-relief annealing. Set $N_f$ is the total life without stress-relief annealing, $N_{fi}$ is the increased life after stress-relief annealing, $a$ is the number of cycles before stress-relief annealing, $a$ is the factor, $a = N_{fi} / N_f$, $0 < a < 1$. The total life of stress-relief annealing is the number of cycles before stress-relief annealing plus the life increased after stress relief, i.e., $N_{fi} = N_f + a^*N_f$.

According to the BCM life model, the double logarithmic coordinate linear fitting of $N_{ia}-N_{ia}$ and $N_{ia}-N_{ia}$ can be obtained:

$$N_{fi} = e^{-1.02 \ln N_f + 19.70}$$

$$N_{fi} = e^{-1.02 \ln (a N_f) + 19.70} + a^*N_f$$

The dispersion belt is used to describe the fatigue reliability of the above model [19]. Test the accuracy of equation (4). As can be seen in figure 5(b), all predicted data are within 1.1 times of the actual test value, indicating that the data accuracy is good and fully conforms to the engineering application.

4.2. Discussion on the influence mechanism of stress-relief annealing on fatigue life

After the fatigue tests were suspended, both the residual stresses and the distortion energy stored in the grain were large. Most residual stresses eliminated by stress-relief annealing, and reduced grain orientation trend. Rearranged or annihilated the dislocations gathered at the grain boundary, so that when retesting, due to the reduction of dislocation obstruction, the deformation of the samples was enhanced. As can be seen from figure 3, stress-relief annealing makes the first stage of the cyclic softening curve decrease greatly, and the response stress entering the second stage is low, which shows that the plastic deformation of the material is easier through stress-relief annealing. Most studies believe that it is related to the decrease of dislocation density [20, 21], residual stress and grain orientation [22, 23]. For example, Xuesong Xiong et al [24] studied the orientation evolution of Fe-Si steel through stress-relief annealing, and believed that stress-relief annealing reduced the percentage of low angle grain boundary and significantly changed the grain orientation. This paper also attempts to illustrate the effect of stress relief on the increase of fatigue life by means of dislocation density and polar diagram.

4.2.1. Dislocation evolution

The variation of the diffraction peak, (110), (200), (211) and (220) are shown in figure 6. At present, the more popular methods for calculating dislocation density are MWH (modified Williamson-Hall) or MWA (modified Warren Averbach) [25, 26]. Dislocation density can be obtained by three times of linear fitting in WMH method, in which the second linear fitting is $H^2=\Delta K$/$D^2/K^2$, where $H^2=(h^2+2h'k'+k'^2)/(h^2+2k'^2+k^2)$. Since martensitic steel obviously has the above four peak shapes, the $H^2$ values of the three planes (110), (211), and (220) are all 0.25, so the error is too large in linear fitting, and the MWA method also needs $\tilde{H}^2(\Delta K-\alpha/D)^2/K^2$ to calculate $\tilde{C}$ value, which is dislocation factor. By amplifying the
diffraction peaks and fitting the peaks shape with Voigt function, the FWHM (full width at half maximum) of each peak was obtained. The change of FWHM of diffraction peak mainly has two factors: grain size and dislocation density, and the basic data used in the calculation of dislocation density above is peak shape data. Therefore, in this paper, the dislocation evolution in fatigue process is explained by two indexes of sub-grain size and FWHM, which are further confirmed by TEM. Pesicka et al. [27] reported that the full width at half maximum (FWHM) of peak measured by XRD was proportional to square root of dislocation density, $\beta \propto \rho^{1/2}$.

From Pesicka’s results, the dislocation density measured by XRD was similar to that measured by TEM. In BCC metal, the $\{110\} \langle 111 \rangle$ and $\{211\} \langle 111 \rangle$ are active slip systems [28], therefore, compare the changes of FWHM of $\{110\}$ and $\{211\}$ diffraction peaks are shown as a function of number of cycles in figure 7(a). FWHM of $\{110\}$ diffusion peak decreased rapidly from the beginning to 30% cycle life, which means that the dislocation density decreases rapidly. This leads to the rapid occurrence of cyclic softening, which corresponds to the first stage of the softening curve in figure 3(a). The second stage of the softening curve is the slow softening stage. There are small fluctuations in FWHM of $\{110\}$ and $\{211\}$ diffraction peaks in this stage, which first increase and then continue to decrease. However, FWHM of $\{110\}$ diffusion peak increases slowly and does not begin to decrease until 70% of the service life. FWHM of $\{211\}$ diffusion peak begins to decrease until 50% of service life. From the whole life cycle, the dislocation density decreases rapidly. This is consistent with the report in the literature [17], that is, because the softening behavior is also related to the changes of dislocation cell structure and lath width. As shown in figure 7(b), the increase of lath width is not significant. However, with the progress of fatigue, it shows an increasing trend. Figure 8 shows the evolution of dislocation morphology. It can be seen from figures 8(a) and (b) that the dislocation distribution gradually decreases from a large number of entanglements during fatigue. At 70% service life, as shown in figure 8(c), there are even blank areas without dislocations in the lath.

For the sample after stress-relief annealing, as shown in figure 7(a), at 30% of the cycle life, FWHM of $\{110\}$ and $\{211\}$ are reduced by stress-relief annealing. FWHM of $\{110\}$ diffusion peak increased at 50%, then reduced at 70%, but FWHM of $\{211\}$ diffusion peak continuous increased. Stress-relief annealing can prolong fatigue life by reducing FWHM, i.e., low dislocation density. It can be seen from figure 7(c) that during fatigue, multiple slip systems in the organization started to form multiple dislocation sources and produced a large number of dislocations. Dislocations gathered at the slip band through slip or climbing, and the same number of
dislocations merged to form new dislocations to reduce distortion energy and formed a high-density dislocation cell or wall. As the fatigue continued, the dislocation cell or walls were further compressed. When the spacing between dislocation walls were small enough, subgrain boundaries were formed. The formation of subgrain boundaries reduced the dislocation density in the subgrains, FWHM increased, and diffraction peaks moved to a small angle. Therefore, in the fatigue process, the size of the subgrain decreased. Stress-relief annealing makes the dislocations originally piled up at the grain boundaries move into the grains. In the subsequent fatigue process, delay the rate of dislocation pile up at the grain boundary and the time for dislocation tangle to form cells or walls. Therefore, although the subgrain size decreases after stress-relief annealing, on the whole, the subgrain size is larger than that of the sample without stress-relief annealing, as shown in figure 7(c).

4.2.2. Grain orientation
It can be seen from the pole figure analysis, as shown in figures 9 and 10, {110} and {211} planes are distributed around the center in all directions, and the distribution is relatively uniform. When cycles reach 30% of the service life, the distribution of {110} plane begins to concentrate, but with the progress of fatigue, the rotation trend of grains does not tend to be consistent, and there is a trend of dispersion, but until 70% of the service life, it still shows the concentrate orientation. Similarly, as shown in figure 10, the distribution of {211} plane is similar to that of {110} plane. Through stress-relief annealing, the trend of the same orientation of {110} and {211} planes are basically eliminated, which is conducive to prolonging fatigue life.
5. Conclusions

Isothermal fatigue tests were conducted in 300 °C for 4Cr2Mo2V die steel. Suspend and conduct stress-relief annealing in the different cycles. Cyclic softening as a function of the number of cycles was investigated in the viewpoint of dislocation structure, FWHM and pole figure. The influence mechanism of stress-relief annealing on fatigue life was discussed. The results are as follows:

(1) The earlier the stress-relief annealing is, the more beneficial it is to increase the fatigue life. When the stress-relief annealing is carried out at 30%, 50% and 70% of the cycle life, the fatigue life is prolonged by 30.27%, 23.84% and 10.24% respectively. The life model after stress-relief annealing is obtained by modifying BCM model, and the accuracy is high;

(2) The dislocation density can be reduced by stress-relief annealing, so that the dislocations originally piled up at the grain boundaries can move into the grains, delay the time of dislocation cell or wall formation, and eliminate the trend of consistent orientation of most grains in the fatigue process, which is the main reason for prolong fatigue life by stress-relief annealing.

Acknowledgments

JIANG Bin and LI Xiaocheng contributed equally to this work.
This work was financially supported by the Guangdong Province Key Area R&D Program in China under grant NO. 2020B010184002.
This work was also supported by Open Project of State Key Laboratory of Advanced Special Steel, Shanghai Key Laboratory of Advanced Ferrometallurgy, Shanghai University (SKLASS 2019-09) and the Science and Technology Commission of Shanghai Municipality (No. 19DZ2270200).

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

Disclosure statement

No potential conflict of interest was reported by the author(s).
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