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Increasing the fatigue limit of a high-strength bearing steel by a deep cryogenic treatment

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Abstract. High-strength steels typically fail from inclusions. Therefore, to increase the fatigue limit of high-strength steels it is necessary to modify the inclusions and/or the surrounding matrix. The goal must be a higher threshold for crack initiation and/or crack propagation. One possibility to reach this goal seems to be a deep cryogenic treatment which is reported to completely transform the retained austenite as well as to facilitate the formation of fine carbides. Therefore, specimens were annealed before or after deep cryogenic treatment, which was carried out with different cooling and heating rates as well as different soaking times at -196°C. Hardness and retained austenite measurements and fatigue experiments were used to evaluate the different sequences of treatments mentioned above. The fatigue limit increases only after some of the sequences. The results show that the soaking times are not relevant for the fatigue limit but it is very important to temper the specimens before the deep cryogenic treatment. Also, repeated deep cryogenic treatments had a positive influence on the fatigue limit.

1. Cryogenic Cooling and Fatigue

High strength steels are often based on martensitic heat treatments so that it is possible that austenite remains besides the martensitic microstructure in the final material. The volume content and the stability of the retained austenite influence the fatigue limit of these steels significantly. Formerly, the cryogenic cooling was primarily used to reduce the content of retained austenite by transforming it into martensite by cooling the material below the secondary martensite start temperature. Unfortunately the higher martensite content often does not increase the fatigue limit due to microcracks, which were created by microstresses caused by the cooling below room temperature. [1, 2]

Recent reports show a significant influence of cryogenic treatments with controlled temperature rates and long soaking times on the wear resistance of tool steels [3]. Also a change in the carbides morphology by the cryogenic treatment is discussed [4, 5].

2. Experimental Setup

The heat treatment of all material states of the hypereutectic carbon steel (1.0 wt.-% C, 1.5 wt.-% Cr, German trade name 100Cr6) starts with austenitisation in a vacuum furnace at 840°C for 20 min followed by quenching to room temperature in oil. After this hardening different treatments of annealing and/or cooling are conducted. Figure 1 gives an overview of the different treatments. Not all of them were applied to every state: annealing is carried out at 180°C for 2 hours in some cases before and/or after deep cryogenic cooling. The deep cryogenic cooling is realized by cooling the specimens down to the temperature of liquid nitrogen (-196°C). The cooling down-process is either done by placing the specimens directly in liquid nitrogen or by a controlled process with a cooling rate of 1 K/min. Heating up is realized with the same rate in the second case. In the first case of fast cooling the specimens are taken out of the nitrogen and directly put on a table at room temperature. The nomenclature of the specimens indicates the elements given in Figure 1. For instance ASC100A is a state which is annealed, slowly cooled down to -196°C, soaked for 100 hours at -196°C, heated up to
room temperature with 1 K/min and finally annealed, again. There was normally only one round of cooling and heating up, except in case of ASC65A, where the sequence was repeated 10 times with a soaking time of 65 hours in total. All heat treatments were carried out with flat fatigue specimens (for details of geometry see [6]).

The specimens were tested in deflection controlled bending fatigue tests. There were about 20 specimens for each state. The fatigue limit for an ultimate number of cycles of $10^7$ was determined by using the staircase method [7]. The hardness was measured by Vickers indentation test. The retained austenite was calculated from X-ray-diffraction by the 6-line-method [8]. All fracture surfaces were observed in SEM and the non metallic inclusions were analyzed regarding their stress intensity factors [9].

![Figure 1. Schematic curve of the elements for the different paths at the heat treatments.](image)

3. Results

3.1. Hardness and content of retained austenite after Cryogenic Treatment
The just hardened state had a hardness of 950 HV 10 and a content of 11.3 vol.-% of retained austenite. Deep cryogenic cooling of the hardened state did not change the hardness but reduced the retained austenite to 9.5 vol.-%. Final annealing reduced the hardness to 825 HV 10 and the content of retained austenite to 8.0 vol.-%.

Hardening and annealing led to 780 HV 10 and 8.5 vol.-% of retained austenite. A second annealing reduced the content of retained austenite significantly but changed the hardness just slightly to 770 HV 10. All variations of cooling between two annealing treatments did not change the resulting hardness values and the content of retained austenite.

3.2. Fatigue results
All states show a wide scatter in the number of cycles to failure (see Fig. 2) caused by the initiation from nonmetallic inclusions which had different morphologies and are located in different distances from the surface. The fatigue limit of the annealed and not cooled state is 940 MPa. A second annealing increases the fatigue limit slightly to 953 MPa. Slow and fast cooling directly after hardening and before annealing reduced the fatigue limit to about 890 MPa, which indicates that the annealing performed directly after hardening and before cryogenic cooling is evidently for higher fatigue limits, while the rate of cooling and heating does not seem to have any influence on the fatigue limit.
Therefore, in two states the specimens were annealed, cooled fast to -196° C, soaked for 100 and 1000 hours and finally annealed after fast heating up to room temperature. Woehler curves show that a longer soaking time increases the fatigue limit by about 7%. Yet, there is still a reduction of the fatigue limit by the fast cooling process, which can be seen by comparing the fatigue limit of state AA and AFC100A. The second one has still a lower fatigue limit than the just annealed state.

Evidently, the best fatigue limits are expected after annealing, slowly cooling, soaking a long time at -196° C, heating up slowly and annealing again. Starting with a soaking time of 24 hours the fatigue limit is increased linearly with the soaking time up to 100 hours. Unfortunately, a further increasing of the soaking time to 1000 hours did not further increase the fatigue limit.

Table 1. Results of different paths of the deep cryogenic treatments: given are the hardness, the retained austenite and the fatigue limits as well as the mean value of $K_{\text{max}}/K_{\text{mod}}$.

| Vickers hardness | retained austenite | fatigue limit | $K_{\text{max}}/K_{\text{mod}}$ |
|------------------|--------------------|---------------|-------------------------------|
| HV 10            | vol.-%             | MPa           |                               |
| only hardened    | 950                | 11.3          | -                             |
| FC100            | 950                | 9.5           | -                             |
| SC100            | 950                | 9.5           | -                             |
| FC100A           | 825                | 8.0           | 888                           | 0.95 |
| SC100A           | 825                | 8.0           | 896                           | 0.84 |
| A                | 780                | 8.5           | 940                           | 1.00 |
| AA               | 770                | 4.0           | 953                           | 1.06 |
| AFC100A          | 770                | 4.0           | 923                           | 1.09 |
| AFC1000A         | 770                | 4.0           | 988                           | 1.01 |
| ASC24A           | 770                | 4.0           | 964                           | 1.08 |
| ASC65A (10 x SC) | 770                | 4.0           | 990                           | 1.15 |
| ASC100A          | 770                | 4.0           | 1015                          | 1.08 |
| ASC1000A         | 770                | 4.0           | 988                           | 1.10 |
3.3. Evaluation of the inclusions at the fracture surface

To exclude a statistical effect caused by different inclusion morphologies of the fractured specimens the stress intensity factors $K_{\text{max}}$ at the critical site of the nonmetallic inclusions were calculated and compared to the expected value $K_{\text{mod}}$ [9]. By this we get information about the load which is applied to the matrix directly at the inclusion by $K_{\text{max}}$. The higher the ratio $K_{\text{max}}/K_{\text{mod}}$ the higher is the fatigue limit in the matrix directly surrounding the inclusion.

The value of 1 for the state A shows that the calculation works quite well for normally heat treated states. The values below 1 for FC100A and SC100A represent the reduced fatigue limit of these states. The second annealing without cooling increases the $K_{\text{max}}/K_{\text{mod}}$ of about 6 %. Additional slow cooling increases $K_{\text{max}}/K_{\text{mod}}$ slightly to 1.08. Surprisingly the state ASC65A shows an increase of 15 %. This is caused by the repeated cooling, soaking and heating up, which was done 10 times for this state.

4. Conclusion

The surroundings of inclusions and their modification are the key factor in determining the fatigue limit. We suppose that microcracks are initiated at these places by deep cryogenic treatments if the following conditions were not taken into account: the annealing directly after hardening is necessary to reduce the thermal eigenstresses around the inclusions, which result from the different thermal expansion coefficients of the inclusion and matrix, and the microstresses resulting from martensitic transformation. In consequence all states which are annealed before cooling exhibit $K_{\text{max}}/K_{\text{mod}} > 1$.

The fast cooling may cause problems due to the thermal stresses up to 530 MPa tensile stresses which are induced between the interior (24°C) and surface (-196°C) of a specimen. They are high enough to induce microcracks.

However, increasing the fatigue limit of a high-strength bearing steel is possible by deep cryogenic cooling. Therefore, it is essential to anneal before and after deep cryogenic cooling and to cool down and heat up with slow cooling rates. Other treatments led to a decrease of the fatigue limit. The soaking time should have no influence since there is no classical diffusion of C in $\alpha$-Fe possible.

The reason for the beneficial effect to the fatigue limit is yet unclear. Possibly, very fine carbides are formed, but investigations with light and electron microscope did not reveal any evidence for this theory.

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