Thermomechanical Fatigue Testing of Dual Hardening Tool Steels

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Hot-work tool steels are exposed to complex interacting cyclic thermal and mechanical loadings. Due to the combination of strengthening via carbides and intermetallic precipitates, dual hardening steels achieve well-balanced mechanical properties in terms of fatigue strength and fracture toughness. Therefore, dual hardening steels have a great potential for hot-work applications. Herein, out-of-phase thermomechanical fatigue tests are used to simulate the loading conditions experienced in hot-work tool steel applications on a laboratory scale. The testing is conducted on Fe–C–Cr–Mo–V and Fe–C–Cr–Mo–V–Ni–Al alloys to compare common 5% Cr and dual hardening hot-work tool steels. The resistance to thermomechanical fatigue is therefore correlated with single or dual hardening. Both alloys experience softening during the fatigue testing. Atom probe tomography investigations reveal coarsening of the secondary hardening precipitates for both alloys. However, the number density of surface cracks is greater for the 5% Cr hot-work tool steel. The dual hardening steel possesses higher resistance to softening and reaches a higher lifetime.

1. Introduction

Hot-work tool steels experience harsh operating conditions, which include complex interacting cyclic thermal and mechanical loadings. The physical and mechanical properties necessary to withstand these loading conditions are high red hardness, sufficient hot yield strength, temper resistance, resistance to thermal shock, and resistance to heat checking.[1] Heat checks, fine surface crack networks, are the major lifetime limiting factor for tools in die-casting applications. This thermal fatigue can either be caused by alternating stresses that develop due to time-dependent temperature gradients, resulting in external loads. These conditions are called “thermomechanical fatigue” (TMF). In the case of position-dependent temperature gradients, no external loads but residual stresses lead to “thermal stress fatigue.”[2] When hot material is injected into a die, the thermal expansion of the tool surface is restrained by the cooler core, resulting in compressive stresses in regions close to the surface. When these stresses exceed the yield strength of the tool material, which decreases with increasing temperature, local plastic deformation occurs, resulting in the formation of residual tensile stresses. During the lifetime of a tool, the accumulated plastic deformation can lead to alternating compressive-tensile stresses with a continuous shift of the mean stress into the tensile region. These loading conditions ultimately lead to the formation of surface crack networks.[1,3,4]

Influencing factors on heat checking resistance include thermal softening, cyclic softening, development of residual stresses, and the microstructure. Thermal conductivity and the coefficient of thermal expansion influence the resistance against heat checking as well. While the former leads to a more uniform thermal expansion and thus indirectly reduces thermal stresses, the latter has a direct influence on thermal strains and thereby thermal stresses.[4–6] In the case of low strain ranges, high tensile strength can increase the lifetime during thermal fatigue, whereas in the case of high strain ranges, ductility is more important, and high strength can have a negative effect on the lifetime.[7] The investigation of different atmospheres during immersion tests of hot-work tool steels in molten Al showed a shift from global oxidation in air and nitrogen to localized oxidation at surface cracks in Ar atmosphere.[8]

Thermal fatigue was tested by Siller via heating hot-work tool steels by laser irradiation.[5] It was observed that the maximum surface temperature has a major influence on the thermal fatigue lifetime of hot-work tool steels. For temperatures below 650 °C, the investigated alloys showed cyclic isotropic softening within the first 5000 cycles, depending on the duration of the thermal loading. After this initial stage, the surface hardness remained
constant, and the materials reached a stable condition. For maximum surface temperatures exceeding 650 °C, no stable conditions were met, but continuous softening occurred, associated with the formation of surface crack networks. It was shown that especially prior austenite grain boundaries act as fatigue crack initiation sites.

Thermal fatigue tests are often performed on hot-work tool steels by cyclic induction heating of internally water cooled hollow cylinders\cite{9,10} or by repeated immersion of internally cooled specimens into a bath of molten Al.\cite{8,11} TMF tests are conducted by inductive heating of solid or tubular specimens, whereas a certain percentage of the thermal expansion is mechanically suppressed.\cite{12} However, in literature no data can be found on the TMF behavior of dual hardening steels.

These dual hardening steels represent a promising alloying concept for hot-work applications. Due to their combination of secondary hardening carbides and intermetallic precipitates, high hardness levels of up to 60 Rockwell hardness, scale C can be achieved, whereas the amount of embrittlement caused by large primary carbides is reduced compared with chromium hot-work tool steels.\cite{13–15}

This study aims to investigate the influence of single and dual hardening on resistance against TMF. Out-of-phase thermomechanical fatigue (OP-TMF) tests have been conducted on a common hot-work tool steel and a dual hardening steel. A test rig developed by Riedler et al.\cite{16} was used for the fatigue testing. The material behavior under TMF loading conditions was analyzed during the TMF testing by recording the stress–strain hysteresis. The amount of softening was evaluated by room temperature hardness measurements of the tested specimens. The influence of microstructure and oxidation behavior was investigated using light optical microscopy (LOM), scanning electron microscopy (SEM), and atom probe tomography (APT).

2. Experimental Section

The chemical compositions of the investigated alloys are shown in Table 1. The dual hardening alloy A was solution annealed at 990 °C for 30 min, followed by oil quenching to room temperature. Ageing was conducted three times at 585 °C for 3 h. The 5% Cr hot-work tool steel, referred to as alloy B, was austenitized at 1050 °C for 30 min, air cooled to room temperature, and subsequently annealed three times at 620 °C for 2 h. Hardness was measured in Vickers HV 5, and the mean value and standard deviation of three measurements were given for every condition.

Strain-controlled OP-TMF tests were performed on samples produced according to Figure 1. An extensometer length of 12.6 mm was used. For temperature control and measurement, three-point temperature measurement using K-type ribbon thermocouples located within 20 mm of the center of the specimen was carried out. The thermocouple that was used for temperature control was placed in the center of the specimen and the second and third about 5 mm below and above the central one. Minimum temperature was 200 °C and maximum temperature was 636 ± 6 °C for alloy A and 641 ± 5 °C for alloy B, with a constant heating and cooling rate of 10 °C s⁻¹ and no dwell time. Specimens were heated with a high-frequency induction system and cooled with pressurized air. At the start of each measurement, a zero stress test was performed to obtain the thermal expansion in the desired temperature range. The mean and maximum longitudinal temperature difference was 4 and 11 °C for alloy A and 7 and 24 °C for alloy B.

As shown in Figure 2, a phase shift of 180° between thermal and mechanical loading was used. The strain amplitude was 0.3% and the ratio between thermal and mechanical expansion −0.91 for alloy A and −0.85 for alloy B. A more detailed description of OP-TMF testing\cite{17} and the test setup used in this investigation can be found in the study by Riedler et al.\cite{16}

TMF lifetime was determined as suggested by Hähner et al.\cite{17} As shown in Figure 3a, TMF lifetime was calculated at a drop of 10% below a tangent line drawn at the last point of zero curvature of the maximum tensile stress.

For microstructural analysis, the TMF-tested samples were cut along the longitudinal axis. Metallographic samples were produced using standard grinding and polishing techniques,\cite{18} with special emphasis on the preservation of the surface oxide layer. Fracture surfaces were investigated with a stereo microscope. Analysis of the TMF-tested microstructure as well as the oxide layer was done with LOM. The 5% Cr hot-work tool steel was etched with 3% HNO₃. Samples of the dual hardening alloy were etched with W2.\cite{18} The sample surface and formed oxides were investigated with an SEM Zeiss Evo50. High-resolution analysis was performed by APT with a LEAP 3000 XHR. Tips were produced electrolytically by standard techniques.\cite{19} In the case of TMF-tested specimens, special care was taken to assure the investigated volume was as close as possible to the fracture surface, i.e., in the area of the most precise temperature control. Due to a high number density of large precipitates, which tend to act as fracture sites, measurements were carried out in laser mode at 60 K. Laser pulse energy was set to 0.2 nJ, and a laser frequency and target evaporation rate of 250 kHz and 0.5% were used, respectively.

![Figure 1. Geometry of the TMF samples, dimensions in mm.](image-url)
3. Results and Discussion

3.1. Thermomechanical Fatigue Testing

The results of the TMF tests are shown in Figure 3. For the dual hardening alloy, 2 separate TMF-tests were performed, called alloy A-1 and alloy A-2. The TMF-test alloy B-1 was performed on the 5% Cr hot-work tool steel. In Figure 3a, the maximum tensile stress of each cycle, recorded at minimum temperature of 200 °C, is shown. Figure 3b shows the evolution of the maximum compressive stress, recorded at maximum temperature. Figure 3c,d show the development of the mean stress and the stress amplitude during TMF lifetime. It can be seen that within the first few cycles, high tensile stresses are reached. Alloy B-1 reaches its maximum tensile stress of 894 MPa after 616 cycles. This fast development of high tensile stresses is caused by the plastic deformation during the heating step and simulates the formation of residual tensile stresses during application. After the peak value is reached, the maximum tensile stress decreases continuously, which is attributed to thermal softening. After roughly 2500 cycles, the maximum tensile stress shows a rapid drop, which is correlated with the growth of macroscopic fatigue cracks.

The dual hardening alloy A-1 reaches its peak tensile stress of 878 MPa at 1869 cycles, and the rate of increase of the maximum tensile stress within the first cycles shows a more continuous behavior. The test of alloy A-2 reaches a significant higher lifetime compared with alloy A-1. The deviation between the two stress signals starts at cycle 330, with alloy A-2 experiencing a less steep change of the stress amplitude and mean stress. This can be attributed to a deviation from the desired maximum testing temperature of 623 ± 10 °C for alloy A-2 and 636 ± 6 °C for alloy A-1. Due to the lower temperature and subsequent mechanical loading, the specimen alloy A-2 takes longer to reach a comparable overaged state, resulting in an increased lifetime. Thus, the fatigue test of alloy A-2 reaches its maximum tensile stress of 844 MPa at 3954 cycles and peak stress amplitude of 497 MPa at 4012 cycles.

Due to the deviations during the tests, further investigations will be limited to the TMF-tests of alloy A-1 and alloy B-1. The slope of the decreasing maximum tensile stress after the maximum value is reached is lower for alloy A-1 than for alloy B-1, indicating a reduced amount of thermal softening. Therefore, alloy A-1 shows a higher TMF lifetime of 4169 cycles compared with 2587 cycles for alloy B-1.

The peak value of the maximum compressive stress shown in Figure 3b is –703 MPa for alloy A-1 and –664 MPa for alloy B-1. Both alloys reach the maximum compressive stress and thus the most severe plastic deformation at cycle number 2. The evolution of the maximum compressive stress is similar to the maximum tensile stress for both alloys. The lowest value before the determined lifetime of the respective alloy is reached is –139 MPa at 1767 cycles for alloy A-1 and -160 MPa at 655 cycles for alloy B-1.

The mean stress in Figure 3c shows a rapid shift into the tensile region, resulting in alternating tensile–compressive loadings. Considering that during the OP-TMF testing the thermal expansion during heating is suppressed, this behavior is indicative of plastic deformation and comparable to the formation of residual tensile stresses. In the evolution of the stress amplitude shown in Figure 3d, softening of both of the investigated materials can be observed. For the first 1200 cycles, the stress amplitude is higher for alloy B-1 and reaches a maximum value of 532 MPa at 616 cycles. The peak amplitude is 511 MPa for alloy A-1 and reached at 1256 cycles. However, the slope of the decreasing stress amplitude is greater for alloy B-1. As the decrease in the stress amplitude is a measure of softening, it is concluded that the dual hardening alloy A possesses a higher resistance against softening in the OP-TMF setup, resulting in the increased lifetime compared with alloy B. This thermomechanical softening is attributed to overaging and coarsening of the microstructure and secondary hardening precipitates.
In Figure 3e, the mechanical stress–strain hysteresis for the first and \( N_{f/2} \)-cycle of the TMF tests alloy A-1 and alloy B-1 are shown. Both alloys experience large plastic deformations within the first cycle. However, the plastic strain is clearly higher for alloy B-1, which according to Manson–Coffin’s law results in a lower fatigue lifetime. With increasing cycle number, the plastic strain amplitude decreases for both alloys. At \( N_{f/2} \), hardly any plastic deformation is detectable.

### 3.2. Microstructural Investigations

In the final heat treatment condition, the microstructures of both materials consist of tempered martensite containing micrometer-sized primary carbides. Alloys A and B reach a hardness of 617 ± 2 HV5 and 654 ± 4 HV5, respectively. After the TMF testing, the hardness of alloy A drops to 363 ± 4 HV5 and 356 ± 2 HV5 for alloy B. Due to the heating rate of \( \pm 10 \text{ K s}^{-1} \) and 6762 cycles until fracture, alloy A was exposed to temperatures exceeding 500 °C for 51.1 h leading to a reduction of hardness by 41%. Alloy B in contrast endured only 2864 cycles and thus temperatures exceeding 500 °C for 21.8 h until fracture, resulting in a hardness drop by 46%. Although alloy A was longer exposed to the increased temperatures, the hardness drop is more pronounced for alloy B, indicating a higher tempering resistance for alloy A.
Siller\textsuperscript{[5]} reported continuous softening of hot-work tool steels if the surface temperatures exceeded 650 °C during thermal fatigue testing, which is slightly higher compared with the findings in this study. However, the testing device used by Siller used laser irradiation inducing a thermal gradient within the tested specimens, whereas the test setup used in this study uses a near constant temperature distribution in the sample volume. This deviation in temperature and stress distribution is therefore likely to be the main cause for these differing observations.

In Figure 4, micrographs of the fracture surfaces are shown. The numerous initiated fatigue cracks are visible as smooth areas along the circumference of the specimens. Because the fatigue testing was conducted in air atmosphere, the entire fracture surface of both alloys is covered by an oxide layer. The metallic bright area at the right of Figure 4b marks the position of the sample fracture.

As can be seen in the cross sections of TMF-tested samples in Figure 5, crack paths are covered by oxide layers as well. Adjacent to large cracks, both alloys show numerous small cracks with a length of a few microns underneath a thick oxide layer. However, the crack density and therefore the amount of the surface area covered by the thick oxide layer are much greater for alloy B. These oxides have a duplex structure, consisting of a thin and compact outer and a thick, porous inside layer. Similar findings were made in the study by Le Roux et al.,\textsuperscript{[10]} where a duplex oxide scale on TMF-tested X38CrMoV5 consisting of an inside layer of $\text{Cr}_2\text{O}_3$ and an outer porous $\text{Fe}_2\text{O}_3$ layer was reported.

Etched cross sections of the fracture surface are shown in Figure 6. In Figure 6a, the martensitic microstructure of alloy A can be determined. In contrast, no martensitic microstructure is visible in the micrograph of alloy B in Figure 6b. Parts of the oxide layer covering the fracture surface are lost during sample preparation. The visible oxide has a reduced thickness compared with the surface oxide layer in close proximity to large fatigue cracks, but shows the same duplex structure. It can be seen that for both investigated alloys, neither fatigue cracks nor the fracture

![Figure 4](image_url)

**Figure 4.** Stereo microscopic micrograph of the oxide covered fracture surface of a) TMF-tested alloy A and b) TMF-tested alloy B.

![Figure 5](image_url)

**Figure 5.** Cross sections of the specimen surface with large cracks and oxide layer of a) alloy A and b) alloy B. Adjacent to large cracks a thick oxide layer is formed, covering small surface cracks.
surface propagates along grain boundaries. However, the fatigue cracks propagate perpendicular to the loading direction.

As shown in Figure 7, numerous fatigue cracks have been initiated on the surface of the specimens. The number density of these surface cracks is greater for alloy B compared to alloy A. These cracks are, in contrast to conventional heat checks, oriented parallel to each other and perpendicular to the longitudinal axis of the specimens. The reason for this behavior is the test setup, where a temperature gradient is simulated by a mechanical constraint of the thermal expansion along the longitudinal axis of the specimen and thus plastic deformation is limited to this direction. A quantification of the crack density was not performed as an unknown number of surface cracks is covered by the thick oxide layer. Especially for alloy B, the high crack density and resulting oxidation leads to an almost complete coverage of the sample surface with thick oxide.

The micrograph of alloy A in Figure 7a shows that only in close proximity to large surface cracks a thick oxide layer has formed. These oxides have a cloudy shape and propagate perpendicular to the fatigue cracks in loading direction. Further investigations showed series of parallel-oriented cracks propagating perpendicular to the loading direction underneath these thick oxide layers, which is also visible in Figure 5. No evidence of material damage caused by circumferential stresses introduced by thermal gradients could be detected in any of the investigated specimens. The specimen surface not covered by thick oxides appears orange for alloy A and rose for alloy B, indicating an oxide layer of a few hundred nanometers thickness. These findings are in contrast to the investigations presented in the study by Le Roux et al., where immersion tests of hot-work tool steels into molten Al were conducted. In air atmosphere, a global oxidation was observed. Only a reduced oxygen partial pressure resulted in similar oxide structures adjacent to large surface cracks, as was also observed in this study.

3.3. High-Resolution Analysis by Atom Probe Tomography

The following results aim to give an impression on the impact of the TMF testing on the microstructure of the investigated steels. However, due to poor statistics, no attempt will be made to quantify the volume fraction, number density, or particle size of the secondary hardening precipitates.

3.3.1. Alloy A

Figure 8 shows the reconstruction of an atom probe measurement of alloy A aged at 585 °C. A high number density of evenly distributed, spherical particles enriched in Ni and Al is visible,

![Figure 6](image1)

![Figure 7](image2)
which form the intermetallic NiAl phase in a size range of a few nm. As can be derived from the proximity histogram of such a particle shown in Figure 8c, only a small Fe content is present within the precipitates. Furthermore, they show an enrichment in Mn and depletion of Cr compared with the surrounding iron matrix. The distribution of carbide forming elements in Figure 8a such as C, Mo, and V seems more irregular compared with that of NiAl. Despite small, plate-like carbides situated in close proximity to NiAl particles, two almost parallel plate-like structures stretching through the entire diameter of the measured volume are visible. The smaller carbides close to the NiAl particles reach a lower carbon content compared with the large structures. Within the plate-like carbide structures, the carbon content varies from 20 to 30 at% C, resulting in the mean core value of 25 at%, as shown in Figure 8d. The carbides are depleted in Ni and Al. If considered that some amount of the carbon is lost due to multiple detector events,\cite{21} it can be assumed that these are $M_2C$ or possibly $M_23C_6$. Prior microstructural investigations of this alloy showed no $M_23C_6$ carbides in this heat treatment condition,\cite{15} which is why it is assumed that these carbides represent an early or transition state of $M_2C$ carbides.

The microstructure of alloy A after TMF testing is shown in Figure 9. It is clearly visible that both precipitate populations are significantly coarsened. The chemical composition of the NiAl particles has not changed compared with Figure 8 and still contains about 10 at% Fe. However, the chemical composition of the detected carbides does differ significantly, which possess a carbon content of 40 and 25 at% of both V and Mo and are thus likely MC carbides.

### 3.3.2. Alloy B

Figure 10a shows the microstructure of alloy B annealed at 620 °C and Figure 10b in the TMF-tested condition. In the heat-treated condition, plate-like carbide structures as well as spherical carbides are visible, similar to alloy A. The carbide plates contain a core carbon content of $\approx 30$ at% C and are considerably enriched in Mo. This could give rise to the assumption that the detected carbides are $Mo_2C$ carbides. After the TMF testing (Figure 10b), a low particle number density of large carbides is present. However, these carbides contain an increased Fe content of up to 10 at%. Significant coarsening of the carbides due to the TMF testing is visible.

It is assumed that the improved tempering resistance and therefore the increased TMF lifetime of the dual hardening alloy is caused by the combination of secondary hardening carbides and intermetallic precipitates. Upon ageing, the intermetallic particles precipitate very fast and homogeneously distributed due to their coherency to the iron matrix,\cite{24} expanding the preferred precipitation sites for secondary hardening carbides from dislocations and grain boundaries to interfaces between the NiAl
Figure 9. APT measurement of TMF-tested alloy A-1. a) From left to right: atom maps of (C, Mo, V) Al and Ni. b) Isoconcentration surfaces of 25 at% (Ni + Al) in green and 5 at% (Mo + V) in pink. c) Proximity histogram according to (Ni + Al) isosurfaces. d) Proximity histogram according to (Mo + V) isosurfaces. Zero distance on the abscissa corresponds to the precipitate/matrix interface; positive values are inside the precipitate.

Figure 10. APT measurements of alloy B. a) Annealed at 620 °C. b) TMF-tested specimen alloy B-1. Left: Atom map of C, Mo, and V. Right: isoconcentration surfaces of 5 at% (Mo + V). Bottom: Proximity histograms according to isosurfaces, zero distance on the abscissa corresponds to the carbide/matrix interface; positive values are inside the carbides.
particles and the iron matrix. This leads to an increased amount of precipitation sites, thus a more homogeneous distribution and size refinement of the secondary hardening carbides, resulting in an increased tempering resistance. Furthermore, the atom probe data shows that in the dual hardening steel, after TMF testing, a higher particle number density is present due to two different populations of precipitates.

4. Conclusions

In this study, out-of-phase TMF tests, reproducing the complex cyclic loading conditions hot-work tool steels experience during application, were performed. The investigated materials include a 5% Cr hot-work tool steel and a dual hardening steel, containing intermetallic NiAl as well as carbide secondary hardening precipitates. The dual hardening alloy showed an increased resistance against softening during the TMF testing, resulting in a higher lifetime and lesser surface crack density compared with the chromium hot-work tool steel.

Following conclusions can be drawn: 1) The TMF testing leads to a shift of the mean stress into the tensile region through plastic deformation. A constant drop of the stress amplitude is visible during the TMF lifetime and is more pronounced for the chromium hot-work tool steel. 2) Numerous fatigue cracks are initiated around the circumference of the specimens. The surface of these cracks is covered by an oxide layer. 3) The dual hardening alloy shows higher tempering resistance and a higher resistance to TMF than the chromium hot-work tool steel. APT was able to reveal significant coarsening of the secondary hardening precipitates in both investigated alloys during the TMF testing.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords

atom probe tomography, dual hardening steels, fatigue testing, heat checking, hot-work tool steel, secondary hardening, thermomechanical fatigue

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