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Influence of non-metallic inclusions on the fatigue properties of heavily cold drawn steel wires.

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Abstract

The influence of non-metallic inclusions on the fatigue properties of heavily cold drawn steel wires is studied. Through a fractography study it is possible to link the inclusion properties, such as chemical composition, geometry, size and location, to the fatigue properties.

Comparing the Stress Intensity Factors (SIF) of non-metallic inclusions to the threshold value for long crack growth shows that the defect size that should be used to calculate the SIF is larger than the inclusion size itself. This is validated using Scanning Electron Microscopy (SEM) in combination with a Focused Ion Beam (FIB). This SEM/FIB study shows that the region around non-metallic inclusions is characterized by alterations in the microstructure that should be taken into account. It is calculated that the SIF increases with 16.5 ± 3.6 % compared to Murakami’s way of calculating SIF of non-metallic inclusions.

Keywords: Steel Wire; Fatigue Crack Initiation; Fatigue Thresholds; Internal Crack; Non-metallic Inclusions; Fractography; Stress Intensity Factor.

1. Introduction

Nowadays heavily drawn steel wires have a wide spread of important industrial applications. In some of these applications the dynamic properties are crucial. Despite the fact that the properties of drawn steel wires changed due to the tendency of producing thinner wires with higher tensile strengths, little research on their dynamic properties was published during the last 10 years.

It is known from research on high strength steels [1-18] that the presence of non-metallic inclusions (further referred to as inclusions) can affect the fatigue properties in a negative way. Several studies on high strength steels show that both the short and long fatigue lives are affected by the presence of inclusions [6,11,15,16,18]. In all cases the short fatigue lives are controlled by surface crack initiations at surface defects and the long fatigue lives are controlled by internal crack initiations at inclusions [1-18]. In the literature on high strength steels the influence of the inclusion size and location is clear, but little research is done on the influence of the inclusion composition and the inclusion geometry on the fatigue properties. Since the inclusion composition and the inclusion geometry have an influence on the stress state in and around the inclusion [19] it is interesting to study the influence of these factors.

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on the fatigue life.

Due to the extensive plastic deformations during the wire drawing process it is not possible to extrapolate the findings on high strength steels to heavily drawn steel wires. Therefore, the influence of inclusions on the fatigue properties of heavily drawn steel wires is investigated in this study.

2. Experimental procedure

2.1. Material

Two different brass coated steel wires are used in this study. The first wire has a diameter of 175 μm and an ultimate tensile strength (UTS) of 3187 ± 37 MPa and was cold drawn to a strain of 3.5. The wire has a threshold stress intensity factor range for long crack growth ($\Delta K_{th,lc}$) of 3.82 ± 0.09 MPa$\cdot$m [20]. The second wire has a diameter of 300 μm and an UTS of 3300 ± 100 MPa and was cold drawn to a strain of 3.6. The wire has $\Delta K_{th,lc}$ of 4.14 ± 0.05 MPa$\cdot$m [20]. Both wires have the same carbon equivalent (%CE = %C + %Mn/6). It is important to note that the wire rod used to draw both wires comes from different suppliers, meaning that differences in inclusion properties (size, composition and distribution) can be expected.

2.2. Fatigue tests

Fatigue tests were performed on a hydraulic Schenk fatigue machine with a linear actuator PLZ 7 and a 1 kN load cell. The experiments done in this study are stress driven pull-pull fatigue tests performed at a frequency of 60 Hz and with a gauge length of 80 mm. The fatigue tests were performed in a controlled environment with a relative humidity of 54 ± 4 % and a temperature of 20.4 ± 0.3 °C.

Two different loading conditions were used for the wires with a diameter of 175 μm. The majority (about 80%) of the tests were done at a stress amplitude ($\sigma_a$) of 707 MPa and a load ratio (R) of 0.5 ($\sigma_{max} = 2827$ MPa). The other wires with a diameter of 175 μm were loaded at $\sigma_a = 1123$ MPa and R = 0.2 ($\sigma_{max} = 2827$ MPa). The wires with a diameter of 300 μm were loaded at $\sigma_a = 670$ MPa and R = 0.5 ($\sigma_{max} = 2680$ MPa).

2.3. Microscopy

A fractography study was performed on all fatigue fractures with scanning electron microscopy (SEM) using a FEI XL30 FEG system. Only the fatigue fractures that failed due to the presence of inclusions are described in this paper.

The microstructure around inclusions is characterized with a SEM/FIB (focused ion beam) using a FEI Dual beam Nova 600 Nanolab system. The material was etched away using a FIB current of 0.92 nA and the microstructure was made visible using FIB imaging with a current of 9.3 pA.

3. Results and discussion

In the first part of this paper (paragraph 3.1) the influence of the inclusion properties on fatigue is described. The influence of the inclusion size, location, chemical composition and geometry is investigated. In the second part of the paper (paragraph 3.2) the stress intensity factor range ($\Delta K$) of the inclusions is compared to $\Delta K_{th,lc}$. Based upon microstructural investigations it is concluded that Murakami’s formulae for calculating the $\Delta K$ value of inclusions [21] can not be used successfully for heavily drawn steel wires.

3.1. Influence of inclusion properties on the fatigue life

3.1.1. Inclusion size and location

The influence of the inclusion size and location can be deduced directly from Murakami’s formulae [21] for surface (eq. 1) and internal (eq. 2) inclusions.
\[ \Delta K_{\text{surface}} = 0.65 \cdot \Delta \sigma \sqrt{\pi \cdot \text{area}} \]  
\[ \Delta K_{\text{internal}} = 0.5 \cdot \Delta \sigma \sqrt{\pi \cdot \text{area}} \]  

\( \Delta K \) is the stress intensity factor range, \( \Delta \sigma \) is the applied stress range and \( \text{area} \) is the area of the inclusion projected on a plane perpendicular to the loading direction. For surface inclusions the weakened area [21] is added to the inclusions projected area.

The \( \Delta K \) value for both surface and internal crack initiations is calculated using the square root of the inclusion size (\( \sqrt{\text{inclusion area}} \)). Since the \( \Delta K \) value is the driving factor for the crack growth rate, there is a tendency of a decreasing fatigue life with an increasing inclusion size. This tendency is also observed for high strength steels [3,5-8,11,13,15-18]. Figure 1 shows the inclusion area of the fatigue fractures as a function of the fatigue life. Although the scatter is large, both wire diameters show a tendency of decreasing fatigue life for an increasing inclusion size. In figure 2 the \( \Delta K \) value is shown as a function of the fatigue life. Since the scatter is decreased by calculating the \( \Delta K \) value, the tendency of decreasing fatigue life with increasing \( \Delta K \) value can be observed more clearly.

![Fig. 1. Inclusion area as a function of the fatigue life.](image1)

![Fig. 2. \( \Delta K \) value calculated with equation 1 and 2 as a function of the fatigue life.](image2)

The influence of the inclusion location (surface or bulk) on the fatigue properties is taken into account in eq. 1 and 2 by the difference in geometrical factor between (0.65 – 0.5). This difference is related to the different boundary conditions between surface and internal fatigue cracks. In figure 1 it can be seen clearly that for a certain inclusion size, the fatigue life of surface crack initiations is significantly lower than for internal crack initiations.

### 3.1.2. Inclusion composition

When the influence of the inclusion composition on the fatigue properties is studied, two different influencing factors can play a role: the difference in thermal expansion coefficient and the difference in mechanical properties between the inclusion and the steel matrix. It is important to note that the difference in thermal expansion coefficient
is not taken into account here because it manifests itself immediately after patenting but the stress state in and around the inclusion will be changed completely due to the extensive plastic deformation caused by the wire drawing production process after patenting. Therefore only the difference in Young’s modulus (ΔE), which is caused by the difference in chemical composition of the inclusions and the steel matrix, will be taken into account. ΔE can have an influence on the fatigue properties because it will influence the stress state in and around the inclusions. Further it is possible that ΔE has an influence on the deformation behavior during the wire drawing production process.

To study the influence of the inclusion composition (and the inclusion geometry) on the fatigue properties only the internal fatigue fractures are investigated in depth because the difference in fatigue life between internal fractures is clearer than for surface crack initiations. Further it is possible that when the fatigue crack initiates at the surface of the wire, environmental influences overshadow the influence of the inclusion properties.

The inclusion composition of all internal fatigue fractures is analyzed with microanalysis and the E modulus is calculated using the rule of mixtures (E\text{inclusion} = E_aV_a + E_bV_b + ...) with E_i the Young’s modulus of oxide i and V_i its volume fraction. Following values of E_i are taken [22-24]: Al_2O_3: 402 GPa, MgO: 300 GPa, SiO_2: 95 GPa, CaO: 215 MPa, MnO: 173 GPa and TiO_2: 287 GPa.

The left side of figure 3 shows a clear correlation between the ΔK value and the fatigue life for all the internal fatigue fractures of both wires. Looking to the microanalysis it is observed that the 175 μm wire loaded at σ_a=1123 MPa and the 300 μm wire loaded at σ_a=670 MPa fractured at inclusions composed of a mixture of MgO, Al_2O_3, SiO_2, CaO, MnO and TiO_2. Only the 175 μm wire loaded at σ_a=707 MPa fractured at inclusions that have a significantly different composition. Therefore these fractures are shown in more detail in the right side of figure 3. For each fracture ΔE and the maximum inclusion stress (ΔE\text{incl,max} see paragraph 3.1.3) are given between brackets. Two different groups of inclusions are observed. The first group contains mixtures of MgO, Al_2O_3, SiO_2, CaO and MnO and has an estimated E modulus between 174 and 201 GPa. The second group is almost completely composed of SiO_2, sometimes with traces of Al_2O_3, CaO and MnO, and has an estimated E modulus between 95 and 106 GPa.

A significant difference in fatigue behavior between both groups is observed (for reasons described in paragraph 3.1.3 the measurement at 3.8E7 cycles is not taken into account). The worse fatigue properties of the SiO_2 inclusions can be explained by the significantly higher ΔE than for the other inclusions. The SiO_2 inclusions are located below the regression line since they have a ΔE that is significantly larger than the average of all fractures (ΔE\text{average}=56 GPa), the other inclusions are located above the regression line since the have a ΔE that is significantly lower than ΔE\text{average}.

3.1.3. Inclusion geometry

It has already been shown that the inclusion geometry has an influence on the fatigue properties for high strength steels [14]. The maximum inclusion stress (ΔE\text{incl,max}) that occurs at internal inclusions is calculated using equation 3 [14]:

\[
\Delta E_{\text{incl,max}} = \frac{1}{\pi} \cdot \frac{1}{2} \cdot \frac{E}{R} \cdot \sigma_a^2
\]

Fig. 3. Left: Overview of all internal fatigue fractures. Right: Internal fatigue fractures of the 175 μm wire loaded at σ_a=707 MPa. The ΔK values calculated with equation 2.
\[ \Delta \sigma_{\text{incl.max}} = 2 \cdot \Delta \sigma \cdot (a/b) \]  

(3)

with \( \Delta \sigma \) the applied stress range and \( a \) and \( b \) respectively the longest and shortest semi-axis of the ellipse that is used to approach the inclusion geometry. Equation 3 shows that \( \Delta \sigma_{\text{incl.max}} \) is determined by \( a/b \), meaning that the more an inclusion approaches the geometry of a perfect circle, the lower \( \Delta \sigma_{\text{incl.max}} \) will be.

Figure 4 shows \( \Delta \sigma_{\text{incl.max}} \) as a function of the fatigue life. Although some correlation between \( \Delta \sigma_{\text{incl.max}} \) and the fatigue life can be observed, the scatter is too high to obtain a good fit of a regression line. Only for the \( \text{SiO}_2 \) inclusions a clear correlation is observed. To be able to indicate the influence of the inclusion geometry on the \( \text{SiO}_2 \) inclusions in a clear way, all influencing factors (inclusion size, location, composition and geometry) are shown together in the right side of figure 3. The influence of the inclusion geometry can be observed quite clearly when the following two fractures are compared: \( (\Delta K = 1.69 \text{ MPa\sqrt{m}}, \Delta E = 115 \text{ GPa}, \Delta \sigma_{\text{incl.max}} = 5356 \text{ MPa}) \) and \( (\Delta K = 1.71 \text{ MPa\sqrt{m}}, \Delta E = 115 \text{ GPa}, \Delta \sigma_{\text{incl.max}} = 3252 \text{ MPa}) \). Since the inclusion size (\( \Delta K \)), the inclusion location and the inclusion composition (\( \Delta E \)) are the same for both fractures; the difference in fatigue life can be addressed completely to the difference in inclusion geometry (\( \Delta \sigma_{\text{incl.max}} \)).

Because a clear correlation is observed between \( \Delta \sigma_{\text{incl.max}} \) and the fatigue life for \( \text{SiO}_2 \) inclusions and there is a significant difference in \( \Delta \sigma_{\text{incl.max}} \) compared to the other fractures, the measurement at 3.8E7 cycles is not taken into account in the analysis in paragraph 3.1.2.
through a surface inclusion. Around the non-metallic inclusion microcracks and alterations in the microstructure can be observed.

3.2. Calculation of the stress intensity factors

When looking at the fatigue crack initiations at surface inclusions in figure 2 it is important to note that more than 1/3 of the inclusions has a $\Delta K$ value that is smaller than $\Delta K_{th,lc}$. Whenever $\Delta K < \Delta K_{th,lc}$ short fatigue cracks have to grow until $\Delta K_{th,lc}$ is reached. For internal fatigue crack initiations it was already shown that the growth of short fatigue cracks results in very high fatigue lives by the formation of a facet area \([6, 13]\). Therefore it can be expected that the fatigue life of these surface crack initiations should also increases significantly. When however the fatigue lives of the surface inclusions with $\Delta K > \Delta K_{th,lc}$ are compared to the surface inclusions with $\Delta K < \Delta K_{th,lc}$ no discontinuity can be observed. This indicates that no short fatigue crack growth occurs, meaning that the $\Delta K$ value of the inclusions is actually larger than $\Delta K_{th,lc}$. Therefore it is expected that Murakami’s way of calculating $\Delta K$ for inclusions can not be applied for heavily drawn steel wires. To validate this assumption the microstructure around surface inclusions is investigated in depth using a SEM/FIB dual beam microscope.

To study the microstructure around inclusions it is not possible to use fatigue fractures since possible microstructural changes can be caused by the fatigue testing itself. Therefore only wires without any load history after the production process are used. SEM is used to scan the surface of these wires for inclusions. Whenever an inclusion is found, sections of about 500 nm are made starting before the inclusion and ending after it. Between each section the microstructure around the inclusion is made visible with the FIB. The sections are made following the wire axis. For each section the depth of the inclusion and the altered microstructure is measured. After all sections are made and analyzed, the data is put back together and the projected area of the inclusion and the altered microstructure is estimated. A schematic representation of this experimental procedure is shown in the left side of figure 5. Since the inclusions present in the 300 $\mu$m wire are significantly larger than the inclusions present in the 175 $\mu$m wire (figure 1), only the 300 $\mu$m wire is investigated to increase the probability of finding surface inclusions.

The right side of figure 5 and figure 6 show FIB images of sections through a surface inclusions. Around the inclusion the microstructure is clearly different from the bulk of the material which shows elongated grains following the wire axis. Also microcracks are observed. The presence of the alterations in the microstructure and the microcracks can be explained by the inhomogeneous deformation of the steel matrix around the inclusion during the wire drawing production process. In total 6 surface inclusions were investigated.

Murakami [21] observed that it is not sufficient to use the inclusion area to calculate $\Delta K$ of surface inclusions.
Therefore Murakami suggested to add a weakened area to the inclusion area. Since the observations on heavily drawn steel wires show that Murakami’s approach underestimates the $\Delta K$ value, adaptations are necessary.

It is important to know that equation 1 is based on Murakami’s formula for a surface crack of arbitrary shape and that inclusions can be modeled as cracks because cracks can immediately initiate due to the high stresses present around them. Since microcracks and alterations in the microstructure are observed around inclusions in heavily drawn steels, it is reasonable to use the projected area of the altered microstructure together with the inclusion projected area in equation 1.

Table 1: Altered microstructure around non-metallic inclusions.

| Inclusion | Inclusion + weakened area | Inclusion + microstructure | (Inclusion + microstructure) x100 / inclusion | Difference in $\Delta K$ (%) |
|-----------|---------------------------|----------------------------|-----------------------------------------------|-----------------------------|
| 5.5       | 5.8                       | 9.5                        | 14.8                                          | 13.1                        |
| 9.9       | 9.9                       | 20.4                       | 19.9                                          | 19.9                        |
| 16.3      | 16.9                      | 31.8                       | 18.1                                          | 17.1                        |
| 23.7      | 23.7                      | 50.6                       | 20.9                                          | 20.9                        |
| 24.6      | 25.2                      | 45.1                       | 16.3                                          | 15.6                        |
| 35.6      | 40.3                      | 64.7                       | 16.1                                          | 12.6                        |
| **Average** | **17.7 ± 2.5** | **16.5 ± 3.6** | | |

Table 1 shows the estimated projected areas of 6 surface inclusions together with the difference between the $\Delta K$ values. It is calculated that the $\Delta K$ value of the inclusion + the altered microstructure is $17.7 ± 2.5$ % larger than the $\Delta K$ value of the inclusion alone. The $\Delta K$ value of the inclusion + the altered microstructure is $16.5 ± 3.6$ % larger than the $\Delta K$ value suggested by Murakami (with the weakened area). Figure 7 shows the $\Delta K$ values of the surface inclusions as a function of the fatigue life when the alterations in the microstructure are taken into account. The $\Delta K$ values in figure 7 are calculated using equation 4.

$$\Delta K_{\text{surface}} = 0.65 \cdot 1.165 \cdot \Delta \sigma \sqrt{\pi \text{area}} = 0.76 \cdot \Delta \sigma \sqrt{\pi \text{area}}$$  \hspace{1cm} (4)

Fig. 7. $\Delta K$ values of the surface inclusions calculated using equation 4 as a function of the fatigue life.

Using equation 4, all fatigue fractures of the 300 $\mu$m wire have a $\Delta K$ value that is larger than, or at least falls within the 95% confidence limits of $\Delta K_{\text{th,lc}}$. Therefore it can be concluded that the suggested adaptations of Murakami’s model (eq. 4) are valid for these 300 $\mu$m wires.

If equation 4 is also used for the 175 $\mu$m wires there are still two fatigue fracture with a $\Delta K$ value that is smaller than $\Delta K_{\text{th,lc}}$ (left side of figure 7). Therefore it is not possible yet to say whether or not equation 4 can be used for other wires with a comparable production process. More research is needed to conclude hereon. Nevertheless it...
should be clear that Murakami’s formula underestimates the ΔK value of inclusions in heavily drawn steel wires because microcracks and alterations in the microstructure are present around inclusions due to the wire drawing production process.

4. Conclusions

The influence of the inclusion parameters, such as the inclusion size, location, composition and geometry on the fatigue properties of heavily drawn steel wires are shown. The inclusion size and location influence the fatigue properties in a direct way and their influence can be calculated using Murakami’s formulae [21] (eq. 1 and 2). These findings were also observed for high strength steels.

It was experimentally observed that the inclusion composition and geometry do have an influence on the fatigue properties of heavily drawn steel wires. Both parameters change the local stress state in and around the non-metallic inclusions. The fatigue life will increase when the difference between the E modulus of the non-metallic inclusion and the steel matrix decreases. This difference in E modulus exists due to the difference in chemical composition between the non-metallic inclusion and the steel matrix. The fatigue life will increase when the inclusion geometry approaches a circle (a/b = 1).

Based on experimental and microstructural observations it is concluded that Murakami’s formula underestimates the ΔK values of non-metallic inclusions in heavily drawn steel wires. For the 300 μm wires used in this study it was shown that when the alterations in the microstructure around non-metallic inclusion are taken into account, the ΔK value increases with 16.5 ± 3.6 % compared to the ΔK value Murakami suggested.

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