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INVESTIGATION OF LOW TEMPERATURE CREEP BEHAVIOUR OF PM STEELS

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Abstract
The automotive industry accounts for almost 70% of the total use of water atomized steel powder for powder metallurgical structural components. Nowadays, such components are increasingly used for high demanding applications, where good tolerances and high mechanical properties are combined. However, it has been found that some PM-steel components at low temperature (100-150°C) and high static loading may experience dimensional instability. Hence, high performance diffusion-alloyed powder grade was investigated for low temperature creep/relaxation at 120°C and 20 kN tensile loading (corresponding to 90% of the yield strength of the material). The materials investigated were sinter-hardened and subsequently tempered at different temperatures. Characterization using different techniques (optical microscopy, dedicated testing, X-ray analysis, hardness testing, etc.) was carried out before and after creep testing and it was revealed that each kind of sample exhibited creep/relaxation behaviour correlated to the tempering temperature. The results were compared to test results of components under similar conditions and a good correlation between the test bars and components were found. Moreover, it was found that selecting proper tempering considerably lowered the creep/relaxation response. Hence, the dimensional instability at high static loading conditions for the studied steels could be reduced.

Keywords: dimensional stability, sintered steels, creep/relaxation, tempering

INTRODUCTION
Powder Metallurgy (PM), due to its low cost and high volume production, is a favoured technique for net-shape manufacturing automotive components. Manufacturing through the PM route is attractive due to its low energy consumption and high raw material utilization. Increasing demands for PM steels used in high performance applications necessitates a need for structurally stable components that, for example, withstand high load at slightly elevated temperature. Examples of such applications are components used for certain applications in heavy duty diesel engines. In those conditions the components are often subjected to high static load (e.g. by clamping) and temperatures around 120-150°C. In the present study, a so-called injector yoke is selected as test component. Detailed investigation revealed that the component showed deformation under high static load at long
exposure time and elevated temperatures. The results from the components were correlated to those of tensile specimens. When operating temperatures are above ambient temperatures, it is found that relaxation or creep phenomena may play a significant role [1-5]. Generally, conventional iron and steel alloy systems can relax or creep at low homologous temperatures (T/Tm < 0.3) at stress levels just below the yield strength [6-8]. The clarification of the parameters, influencing this kind of low temperature creep and relaxation, will open wide range of opportunities for PM components in automotive industry.

It is highly important to design PM components with high dimensional stability for high loading applications. For such applications, diffusion-alloyed materials (e.g. diffusion bonding of Ni and Cu to pre-alloyed Fe-Mo steel powder) are most commonly used. A commercial grade of this alloy system produced by Höganäs AB is Distaloy HP, where HP stands for high performance.

In this study, the approach is to use standard tensile bars of Distaloy HP+0.6% C, sinter-hardened and post-sintering treated (tempered) at different conditions, subsequently subjected to creep testing under controlled conditions (stress, time and temperature). The results are correlated with components tested under service-like conditions. Low temperature creep experiments were carried out at high constant tensile loads at temperatures in the range 120-150°C. The creep/relaxation was investigated using samples of different variants of sinter-hardening and tempering. In this way, the effect of tempering on the microstructure and possible correlation with resulting creep or relaxation was studied with the aim of understanding how to minimize creep deformation at high static loads.

**MATERIALS AND EXPERIMENTAL PROCEDURE**

Distaloy HP+0.6% C tensile samples (according to standard ISO 2470) were provided by Höganäs AB, Sweden. The base powder was admixed with 0.6 wt. % graphite as carbon source and 0.6 wt. % Kenolube as lubricant and it was compacted to green density of ~7.2 g/cm³. The specimens were sintered at 1120°C in 90% N₂/10% H₂ atmosphere and sinter-hardened at a cooling rate of between 2.5 and 3°C/s. Tempering was subsequently done at 200 and 300°C in air for 1 hour. Processing data are summarized in Table 1. Distaloy HP+0.6% C engine components (injector yokes) provided by Volvo AB were also sinter-hardened with post hardening treatment performed in the different ways shown in Table 1.

Tab.1. Tensile samples and injector yoke components detail and the processing conditions.

<table>
<thead>
<tr>
<th>Sample denomination</th>
<th>Tensile samples</th>
<th>Injector yokes</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>DHP-UT</td>
<td>DHP-T200</td>
</tr>
<tr>
<td>Tempering in air for 1 hour</td>
<td>No</td>
<td>200°C</td>
</tr>
</tbody>
</table>

( *Except Y-T200 which is sintered + hardened as a separate process)

Tensile testing was carried out at Höganäs AB. Low temperature tensile creep experiments were performed by electro-mechanical universal testing system Instron 5500R with 4505 load frame equipped with an Instron 3119-407 type environment chamber. The creep experiments on tensile samples were performed at constant 20 kN load (equals 90% of the yield strength of the material) as discussed in [1, 2] and the strain change was measured with respect to time.

A special method was developed for investigation of creep/relaxation behaviour of yokes which are subjected to compressive pre-stress while exposed to 120-130°C for
prolonged time. This is virtually very close to the conditions of the tensile test samples. A series of yokes were placed in a centre rod and prestrained to approx. 50 kN at room temperature and then the load was released to measure any dimensional change. The overall height of the yokes before and after pre-stressing was measured by using a special attached fixture with a dial indicator. Then the series of yokes were stressed to a constant load of approximately 35 kN for 90 min at room temperature to observe if there is any initial deformation. Later the same set of components were loaded with approximately 35 kN and placed in a furnace at 120-130°C for a specific time (up to $10^6$ sec).

![Fig.1](image)

**Fig.1.** Standard tensile test specimens at different conditions (a), injector yoke component (b) injector yoke sketch showing the loading direction in bending mode (c).

XSTRESS 3000 equipment was used to measure the austenite content in the tensile samples and injector yokes. The apparent hardness (HV10) and microhardness (HV0.1 & 0.05) measurements were done on WolpertDia Tester and StruersDuraScan 70 testing equipment, respectively. Light microscopy (Leica DMRX optical microscope) and high resolution scanning electron microscopy (LEO 1550 Gemini) were used for metallographic investigations.

**RESULTS AND DISCUSSION**

Based on the tensile results for the Distaloy HP material processed at different conditions (Table 2), the minimum yield strength ($R_{p0.2}$) among all the samples was considered for creep test loading. The sinter-hardened- and un-tempered sample (DHP-UT) exhibits the lowest yield stress and therefore 90% of its yield strength was chosen as the load for creep testing, which means about 20 kN load for the tensile samples.

<table>
<thead>
<tr>
<th>Samples</th>
<th>$R_{p0.2}$ [MPa]</th>
<th>St. dv [MPa]</th>
<th>$R_m$ [MPa]</th>
<th>St. dv [MPa]</th>
<th>Elongation [%]</th>
<th>St. dv [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>DHP-UT</td>
<td>691</td>
<td>50</td>
<td>926.6</td>
<td>23</td>
<td>0.46</td>
<td>0.05</td>
</tr>
<tr>
<td>DHP-T200</td>
<td>807</td>
<td>48</td>
<td>1163</td>
<td>43</td>
<td>0.92</td>
<td>0.19</td>
</tr>
<tr>
<td>DHP-T300</td>
<td>853</td>
<td>36</td>
<td>1025</td>
<td>31</td>
<td>0.53</td>
<td>0.09</td>
</tr>
</tbody>
</table>

$R_{p0.2} = 0.2\%$ offset yield strength, $R_m =$ ultimate tensile strength

As shown in earlier studies [1,2], tensile samples exhibit microstructure that is typical for diffusion bonded powder material (Fig.2). Higher tempering temperature (300°C) results in a typical tempered microstructure, characterised by the beginning of transition carbide precipitation from the martensite.
In order to study the influence of application temperature at a constant load, a short-term (24 h) creep test was done at different temperatures. Figure 3 shows cyclic creep test data of DHP-UT and DHP-T200 with stepwise increases of the holding temperature involving 30°C increments each with 24 h duration. It is clearly seen that in case of DHP-UT at 30°C the strain rate is almost negligible. With increasing holding temperature from 60°C and above, there is an increase in strain up to 90°C and rapid increase in strain at 120°C and onwards. Similar behaviour in case of DHP-T200 was observed, also indicating rapid increase of strain rate at 120°C.

Based on the results from the short-term creep testing at different temperatures, it was decided to perform the long-term creep tests on the samples at constant temperature of 120°C and at constant load of 20 kN.

Figure 4a shows strain vs. time plots of the sinter-hardened and subsequently tempered samples DHP-UT, DHP-T200 and DHP-T300. The data shown are from the prolonged creep testing performed at a constant load of 20 kN at 120°C. The results of the test bars show a clear similarity with the results obtained from the injector yokes (Fig.4b) subjected to a constant force at 120-130°C for prolonged time. The deformation is clearly increasing over time with decreasing prior tempering temperature applied.
Fig. 4. Creep curves (strain vs. time) of the tensile samples tested for 1 million seconds (a), injector yokes dimensional change plot (b), parameter: overall height, measured over at certain periodic interval, dimensional change vs. log(t).

All the samples exhibit the initial stage of creep, i.e. primary or transient creep. The initial increase of strain upon loading until maximum load is constant is also termed instantaneous strain. The DHP-UT samples exhibit primary creep stage for up to 20-25 h and then reach the secondary stage of creep, where the strain rate is steadily increasing. The DHP-T200 samples pass the primary creep at an early period of time and reach secondary creep stage faster and further maintain constant strain rate at this stage. The DHP-T200 samples have a lower strain rate during secondary stage of creep, as compared to the un-tempered samples (DHP-UT). In the case of the DHP-T300 samples, tempered at higher temperature, they possess the lowest change in strain. The strain is stable even up to 1 million seconds (~277 hours) at 120°C. In case of the components, in order to record the primary creep, the tests were carried out for up to 90 minutes and the change in dimensions was measured. The tests were prolonged for one million seconds and again the dimensional change was measured and the results show a similar trend as the tensile samples with respect to the influence of tempering temperature. The stability of the Y-T200 condition during, up to 90 minutes hold, is due to Y-T200 component not being sinter-hardened. It is sintered and then hardened (oil quenched) in a different process.

Fig. 5. Strain rate values of the corresponding tensile samples calculated from the secondary creep of long term creep tests.
The strain rate plot of the respective tensile samples, as shown in Fig. 5, exhibits decreasing strain rate with increasing tempering temperature at high static loads. These results show that dimensional stability can be controlled by proper selection of tempering conditions.

CONCLUSIONS

The creep/relaxation phenomena observed for the components studied are easily simulated by means of controlled testing. Results for diffusion-alloyed sinter-hardened PM steel clearly show the correlation between the tempering temperature and the creep/relaxation behaviour. Significant creep/relaxation was shown for un-tempered and less tempered material (200°C) at 120°C and high loading conditions, while more fully tempered material (300°C) appeared much more resistant. Creep, or relaxation, at moderate temperatures (i.e. 100-150°C) of components subjected to high static load could potentially cause a problem during service.

By selecting a proper heat treatment, however, with a special emphasis on the tempering temperature, the effects of low temperature creep/relaxation can be diminished. To solve this and allow use for applications running at the slightly elevated temperatures of interest (100-150°C), appropriate heat treatment in terms of increased tempering temperature can be adopted to optimize the microstructure and thus minimize the creep or relaxation effect.

Acknowledgement

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