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Microstructural, Mechanical and Fatigue Properties of Cobalt Alloys

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Introduction - General goals

- **reduction** of fuel consumption and pollutant emission
  - higher **efficiency** motor development
  - increase of **fuel injection pressure** in cylinders
  - higher **stresses** in injection system components

\[ \downarrow \]

- inadequacy of steels $\rightarrow$ use of **cobalt based alloys** (Stellites) for components mechanically stressed at high temperature

\[ \uparrow \]

- **literature about Co alloys** mainly concerned on wear and corrosion resistance at high temperature

- **few data about high temperature fatigue** available
Materials & specimens

Tensile and fatigue cylindrical (not notched) specimens, 8 mm diameter
- “stellite 6” type alloy produced by casting
- “stellite 6” type alloy produced by powder metallurgy + hipping

Experimental methods

Mechanical tests
- hardness and micro-hardness test at R.T.
- tensile tests at R.T., 250 or 500 °C
- pulsed traction fatigue tests (R ≈ 0) up to $2 \cdot 10^6$ cycles at 250 or 500 °C

Crystallographic and micro-structural tests
- both on as received material, and after 250 or 500 °C treatments
- X ray diffraction (Co anode)
- optical and electronic metallography and EDS micro-analysis

Fractography
### Chemical composition of cast or HIP stellite grade 6. (% wt.)

<table>
<thead>
<tr>
<th></th>
<th>Co</th>
<th>C</th>
<th>Cr</th>
<th>W</th>
<th>Ni</th>
<th>Si</th>
<th>Mn</th>
<th>Fe</th>
<th>V</th>
<th>Nb</th>
</tr>
</thead>
<tbody>
<tr>
<td>CAST</td>
<td>bal.</td>
<td>1.19</td>
<td>25.54</td>
<td>5.21</td>
<td>1.99</td>
<td>1.56</td>
<td>0.69</td>
<td>0.85</td>
<td>0.028</td>
<td>0.034</td>
</tr>
<tr>
<td>HIP</td>
<td>bal.</td>
<td>1.48</td>
<td>27.21</td>
<td>4.78</td>
<td>0.30</td>
<td>1.21</td>
<td>0.21</td>
<td>0.44</td>
<td>0.021</td>
<td>0.002</td>
</tr>
</tbody>
</table>
Cast sample XRD Analyses (Brag-Brentano geometry, Co anode)

- Shallow and partially overlapped peaks (e.g. 40-65° range)
- Possible phases: $\text{Co}_{\text{CFC}}$, $\text{Co}_{\text{HCP}}$, $\text{Co}_{25}\text{Cr}_{25}\text{W}_{8}\text{C}_{2}$, $\text{Cr}_{7}\text{C}_{3}$, $\text{Cr}_{23}\text{C}_{6}$, $\text{Cr}_{3}\text{C}_{2}$, $\text{Co}_{3}\text{W}_{3}\text{C}$, $\text{Co}_{4}\text{W}_{2}\text{C}$, $\text{Co}_{6}\text{W}_{6}\text{C}$, $\text{W}_{2}\text{C}$, $\text{Co}_{3}\text{W}$, $\text{Co}_{7}\text{W}_{6}$
- Probable prevalence of $\text{Co}_{\text{CFC}}$ in respect to $\text{Co}_{\text{HCP}}$
- Possible phase evolution on heating at 250 or 500°C
Hipped sample XRD Analyses (Brag-Brentano geometry, Co anode)

- Shallow and partially overlapped peaks (e.g. 40-65° range)
- Possible phases: Co\textsubscript{CFC}, Co\textsubscript{HCP}, Co\textsubscript{25}Cr\textsubscript{25}W\textsubscript{8}C\textsubscript{2}, Cr\textsubscript{7}C\textsubscript{3}, Cr\textsubscript{23}C\textsubscript{6}, Cr\textsubscript{3}C\textsubscript{2}, Co\textsubscript{3}W\textsubscript{3}C, Co\textsubscript{4}W\textsubscript{2}C, Co\textsubscript{6}W\textsubscript{6}C, W\textsubscript{2}C, Co\textsubscript{3}W, Co\textsubscript{7}W\textsubscript{6}
- Probable prevalence of Co\textsubscript{CFC} in respect to Co\textsubscript{HCP}
- No phase evolution on heating at 250 or 500°C
Cast samples micro-structure

Main primary dendrites

Inter-dendritic carbides (mainly lamellar form)

As received, 456 x 362 μm

No microstructural differences due to treatment at 250 and 500 °C are noticeable

After treatment at 500 °C, 456 x 362 μm
Co rich matrix, dispersed carbides, about 2 µm diameter. Grain size in the range of 5-40 µm with the most part in the range 5-10 µm.

Larger grains are richer in Co (EDS analysis).
Cast sample micro-analysis (EDS)

Metallic matrix

<table>
<thead>
<tr>
<th>Cr</th>
<th>Co</th>
<th>W</th>
<th>Mo</th>
<th>Si</th>
</tr>
</thead>
<tbody>
<tr>
<td>24</td>
<td>71</td>
<td>3.5</td>
<td>0.24</td>
<td>0.65</td>
</tr>
</tbody>
</table>

Cr rich carbides

<table>
<thead>
<tr>
<th>Cr</th>
<th>Co</th>
<th>W</th>
<th>Mo</th>
</tr>
</thead>
<tbody>
<tr>
<td>78</td>
<td>15</td>
<td>6.3</td>
<td>0.43</td>
</tr>
</tbody>
</table>

Co, W rich carbides

<table>
<thead>
<tr>
<th>Cr</th>
<th>Co</th>
<th>W</th>
<th>Mo</th>
</tr>
</thead>
<tbody>
<tr>
<td>21</td>
<td>47</td>
<td>29</td>
<td>2.7</td>
</tr>
</tbody>
</table>

Back-scattered electrons image
Hardness and micro-hardness

<table>
<thead>
<tr>
<th>Samples</th>
<th>HV 50</th>
<th>HV 0.05 Dendritic zones</th>
<th>HV 0.05 Carbides rich zones</th>
</tr>
</thead>
<tbody>
<tr>
<td>Room temperature</td>
<td>370</td>
<td>400-430</td>
<td>530-1100</td>
</tr>
<tr>
<td>Heating at</td>
<td>395</td>
<td>390-410</td>
<td>550-1000</td>
</tr>
<tr>
<td>Heating at</td>
<td>385</td>
<td>380-400</td>
<td>800-1100</td>
</tr>
</tbody>
</table>

low heat treatment effect on hardness of matrix

Cast sample: scattered results on precipitated carbide zone (hardness indent large in respect to dimension of carbides)

The hardness tests effected on the transverse section of the HIP samples reveal 660 HV for the 50 g tests and 460 HV for the 50 kg tests.

As received cast sample, 92 x 69 μm
Mechanical tests

Tensile test

\( s \text{ [MPa]} \)

\( e \)

- C RT
- C 250°C
- C 500°C
- H 250°C
- H 500°C
Fatigue tests:
- pulsed traction fatigue $R \approx 0$
- staircase method up to $2 \cdot 10^6$ cycles, 30 Hz, temperature 250 or 500 $^\circ$C

Results:
• the highest fatigue resistance was observed for HIP samples
• there is a great difference in the behaviour of cast and hipped samples
• results obtained through 250 or 500 $^\circ$C tests are similar but the best behaviour has been observed at the lower temperature
• under the maximum stress the fracture occurred after a greater cycle number for the samples tested at the lower temperature
Mechanical tests

Cast tensile test sample (no necking).

Hipped tensile test sample (shear lips presence)
Fractography – cast sample tensile test fracture at 500 °C

Mainly inter-dendritic fracture
and
trans-dendritic quasi-cleavage fracture
Fractography – cast sample fatigue test at 250 C

Fracture nucleation from a shrinkage defect
Fractography – cast sample fatigue test at 500°C

Nucleation and propagation fatigue fracture zones

detail of stair-step fatigue propagation
Fractography – HIP sample tensile test fracture at 500 °C

The fracture is ductile, nucleated by the presence of an inclusion
Fractography – hipped sample fatigue test at 500 °C

Fracture surface observed by means of Stereo Macro-scope.

The fatigue fracture is nucleated by the presence of a defect constituted by an Al-Mg oxide.
Fractography – hipped sample fatigue test at 500 °C

Fatigue propagation zone

Final fracture zone characterized by ductile morphology
Discussion and conclusions (I/II)

☆ Cast samples are constituted by cobalt rich primary dendrites and lamellar inter-dendritic zones (eutectic mixtures) with high carbides content.

☆ Dendritic matrix is constituted by a solution of the alloying elements in CFC Co. EDS micro-analyses have evidenced two carbide types: one with high Cr content, the other with high W content.

☆ Hipped samples present a Co rich matrix and dispersed carbides, about 2 μm diameter. Grain size is in the range of 5-40 μm with the most part in the range 5-10 μm.

☆ XRD analyses have shown shallow and partially overlapped peaks (e.g. 40-65 range) with the probable prevalence of $\text{Co}_\text{CFC}$ in respect to $\text{Co}_\text{HCP}$ and the presence of mixed carbides. The cast sample show a phase evolution changing the test temperature, not observed for hipped samples.
Discussion and conclusions (II/II)

- The best performance both in tensile tests and in fatigue tests was observed for the hipped samples.

- The effect of temperature on tensile and fatigue properties is limited; particularly, the observed fracture mechanisms do not change varying the temperature between 250 and 500 °C.

- In tensile test samples the fracture of cast samples is mainly inter-dendritic and only in some zones the fracture are completed by a quasi cleavage inter-dendritic fracture. In hipped samples the ductile mode fracture is nucleated by an inclusional defect.

- In fatigue tests, the crack of cast samples is nucleated by casting defects and propagates on crystallographic planes, in a trans-dendritic way, with a stair-step morphology. The crack of hipped samples is nucleated by an inclusion and the fracture is mainly ductile.
Fractography – tensile test fracture at 500°C (II/II)

quasi-cleavage
Mechanical tests

**Tensile tests cast sample:**

- Pulsed traction fatigue $R \approx 0$
- Staircase method up to $2 \cdot 10^6$ cycles, 30 Hz, temperature 250 or 500°C
- Results obtained through 250 or 500°C tests are similar

**Fatigue tests:**

- Pulsed traction fatigue $R \approx 0$
- Staircase method up to $2 \cdot 10^6$ cycles, 30 Hz, temperature 250 or 500°C
- Results obtained through 250 or 500°C tests are similar
**CAST Co-Alloy**
pulsed traction fatigue tests \((R \approx 0)\), up to \(2 \cdot 10^6\) cycles, at 500 C

<table>
<thead>
<tr>
<th>Strength (Mpa)</th>
<th>Specimens results</th>
<th>Results</th>
</tr>
</thead>
<tbody>
<tr>
<td>450</td>
<td>X</td>
<td>1</td>
</tr>
<tr>
<td>440</td>
<td></td>
<td></td>
</tr>
<tr>
<td>430</td>
<td></td>
<td></td>
</tr>
<tr>
<td>420</td>
<td></td>
<td></td>
</tr>
<tr>
<td>410</td>
<td>X, X</td>
<td>2</td>
</tr>
<tr>
<td>400</td>
<td></td>
<td>1</td>
</tr>
<tr>
<td>390</td>
<td>O, X, O, X</td>
<td>3, 2</td>
</tr>
<tr>
<td>380</td>
<td></td>
<td>1</td>
</tr>
<tr>
<td>370</td>
<td>O</td>
<td>1</td>
</tr>
</tbody>
</table>

X: specimen broken before \(2 \cdot 10^6\) cycles  
O: specimen completes \(2 \cdot 10^6\) cycles
## CAST Co-Alloy

Pulsed traction fatigue tests (R ≈ 0), up to $2 \cdot 10^6$ cycles, at 250 °C

<table>
<thead>
<tr>
<th>Strength (Mpa)</th>
<th>Specimens results</th>
<th>Results</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mpa</td>
<td>1  2  3  4  5  6  7  8  9  10  11</td>
<td>X  O</td>
</tr>
<tr>
<td>450</td>
<td>X</td>
<td>1</td>
</tr>
<tr>
<td>440</td>
<td></td>
<td></td>
</tr>
<tr>
<td>430</td>
<td>X  X</td>
<td>2</td>
</tr>
<tr>
<td>420</td>
<td></td>
<td></td>
</tr>
<tr>
<td>410</td>
<td>X  O  X</td>
<td>2  1</td>
</tr>
<tr>
<td>400</td>
<td></td>
<td>1</td>
</tr>
<tr>
<td>390</td>
<td>O  X  O</td>
<td>1  2</td>
</tr>
<tr>
<td>380</td>
<td></td>
<td>1</td>
</tr>
</tbody>
</table>

X: specimen broken before $2 \cdot 10^6$ cycles  
O: specimen completes $2 \cdot 10^6$ cycles
**HIP Co-alloy**

pulsed traction fatigue tests ($R \approx 0$), up to $2 \cdot 10^6$ cycles, at 500°C

<table>
<thead>
<tr>
<th>Strength (Mpa)</th>
<th>Specimens results</th>
<th>Results</th>
</tr>
</thead>
<tbody>
<tr>
<td>740</td>
<td>X</td>
<td>1</td>
</tr>
<tr>
<td>720</td>
<td></td>
<td></td>
</tr>
<tr>
<td>700</td>
<td>X</td>
<td>1</td>
</tr>
<tr>
<td>680</td>
<td>X</td>
<td>O</td>
</tr>
<tr>
<td>660</td>
<td>O</td>
<td>O</td>
</tr>
</tbody>
</table>

X: specimen broken before $2 \cdot 10^6$ cycles  
O: specimen completes $2 \cdot 10^6$ cycles