SURFACE TREATMENT OF TITANIUM ALLOYS FOR FRICTION AND FATIGUE PROPERTIES
D. Treheux, L. Vincent, Ecole Centrale de Lyon, B.P. 163, 69131 Ecully cedex.

Introduction

Ti-6Al-4V alloys are widely used and many papers consider their fatigue and tribological properties. It is well known that the complex microstructures of Ti-6Al-4V alloys have a significant effect on fatigue cracking and that friction properties are very bad. Moreover any improvement in wear strength by a surface treatment leads to a strong decrease in fatigue limits.

This paper considers surface treatments of a Ti-6Al-4V alloy that may enhance wear strength without decreasing fatigue properties; such a study needs to take into account:
- microstructures through α (hcp) and β (bcc) phases,
- possible phase transformations during cooling or during straining (α′ hcp martensite),
- mechanism of the initiation and of the growth of fatigue cracks.

Surface treatments have been chosen to describe specific surface behaviours and not to give a classification. They are of three kinds:
- hardening by a cold-working
- hardening of a solid solution
- superficial coating (chemical combination, hard layers,...)

Preliminaries

Wear behaviour, jamming strength and fatigue properties were studied by means of a plane-ring tribometer (1), a grips-cylinder Faville machine (1) and at last rotating beam apparatus. Specimens for Faville and fatigue tests were annealed, cut from 8 or 14 mm diameter rods and then surface treated.

Planes for tribometer tests are 12 mm squares in Ti-6Al-4V alloys and rings are made of tempered steels (40 HRC). Tests are performed in water, under an applied load of 400 N and at a speed of 300 rpm. Each specimens are diamond paste polished before superficial treatments. Faville tests are also performed in water, with the following loading schema: 14 mm under a 350 N applied load and 14 mm under 700 N, at a speed of 176 rpm. Pieces were mechanically polished (800 grade paper). Tribometer tests were of a particular use so to analyse chemical changes in the superficial layers. For Faville tests, loss of weight was used as a criterion for wear. Fatigue tests were run at 100 Hz and a specimen was considered as unfailed when it reaches 10⁷ cycles without a catastrophic failure. For each test, specimens annealed before cutting were taken as a reference.

First treatments are summarised in table I. Table II indicates results of Faville tests. The main waited result is a great improvement for all treatments. However differences are noticed following cylinder or grips have been treated. The same tendencies are obtained for plane-ring tests but improvement is greatest for heat treatments with nitrogen that is (H2-N2) annealed or ionic nitriding. For instances, 2 hours testing under 500 N gives a very small loss of weight (less than 2 mg) for nitrogen treated specimens but immediate jamming
Table I: Surface treatments used with the preliminary tests.

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Field of Treatment</th>
<th>Superficial Chemical Gradient</th>
<th>Macroscopic Compact Surface Layers (&gt;1 μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A. annealed</td>
<td>α + β</td>
<td>no</td>
<td>no</td>
</tr>
<tr>
<td>B. annealed in (90 H₂-10 N₂)</td>
<td>α + β</td>
<td>yes</td>
<td>no</td>
</tr>
<tr>
<td>C. oxidizing</td>
<td>α + β</td>
<td>yes</td>
<td>no</td>
</tr>
<tr>
<td>D. borizing</td>
<td>α + β</td>
<td>no</td>
<td>Ti B</td>
</tr>
<tr>
<td>E. ionic nitriding</td>
<td>β</td>
<td>no</td>
<td>Ti₂ N</td>
</tr>
</tbody>
</table>

Table II: Loss of weight for Faville tests.

<table>
<thead>
<tr>
<th>Loss of weight</th>
<th>HEAT TREATMENT</th>
</tr>
</thead>
<tbody>
<tr>
<td>in mg</td>
<td>A</td>
</tr>
<tr>
<td>treated cylinder immediate</td>
<td>89</td>
</tr>
<tr>
<td>treated grips jamming</td>
<td>81</td>
</tr>
<tr>
<td>total</td>
<td>170</td>
</tr>
<tr>
<td>treated cylinder immediate</td>
<td>49</td>
</tr>
<tr>
<td>annealed grips (A) jamming</td>
<td>107</td>
</tr>
<tr>
<td>total</td>
<td>156</td>
</tr>
</tbody>
</table>

occur for untreated specimens tested under 50 N.

Results of fig. 1 indicate that A annealing gives the best fatigue limit and quick "a priori" conclusions may be that:

- hard layers have a strong detrimental effect
- coarse microstructures give poor behaviours. Futhermore M.E.B. observations show that first cracks initiate from cutting striations. It is important to notice that cutting striations are always observed after the several superficial treatments. However we can think that it becomes less harmful because of a diffusion process that decreases notch effects. Thus surface geometry is an important parameter but it does not seem that it is a main one to explain the different behaviours.
In fact important differences are about residual stresses since cutting precedes heating for B, C, D and E treatments. Thus residual stresses due to cutting disappear and we only get the ones caused by cooling without any phase transformation.

Comments and results
To get a better understanding of the main parameters to be taken into account, we then limited this study to one element added in the superficial layers that is nitrogen. Nitrogen gives a thick layer of Ti₂N nitrides (ionic nitriding) or a solid solution (gaseous nitriding). Fatigue and wear results are analysed from
- residual stresses induced by shot-peening or machining
- superficial chemistry
- heat treatments in the matrix and ageing.

Each treatment in Δ + β or in α fields decreases the fatigue limit of as treated specimens and figure 2 shows similar results for thick combination layers obtained during ionic nitriding and for annealing at 850°C under Argon (F, fig. 2). This last treatment induces a very low superficial pollution controlled by glow discharge optical spectroscopy GDOS (2).
Gaseous nitriding in $\alpha + \beta$ field induces a complex superficial chemistry (fig. 3) and a strong decrease in fatigue limit (fig. 2) but a good wear strength (table II). However, room ageing gives an improvement and for one month ageing, fatigue limit increases towards the one of argon treatment. We then notice the desorption of hydrogen from the superficial layers and we suggest as hypothesis, a precipitation of nitrides that may develop residual stresses. Overmore, for friction tests, GDOS analyses indicate a great instability of these surface layers. (fig. 4 and fig. 5)

Another important result is the consequence of a shot-peening on several superficial layers. We always increase the fatigue limit towards the one obtained for annealed pieces (A). Particulary, for ionic nitriding and after shot-peening, a nitride layer is always present but nitrides are broken and this should be considered to have a detrimental effect for fatigue. This result well agrees with the important beneficial effect of residual stresses even with the presence of hard superficial layers.

All these treatments induce an improvement in friction strength with regard to annealed pieces. However, little scars due to nitrides broken by a shot-peening, decrease wear properties.

At last, a machining on pieces gaseous nitrided at 850°C, by a treatment identical to B treatment, gives back a high fatigue limit (fig. 2) and
thus is another prove of the effect of residual stresses in the superficial layers.

Fig. 3: GDOS profile of gaseous nitrided alloys in $\alpha + \beta$ field.

Fig. 4: GDOS profile of N after testing with the tribometer under 400 N.
Experiments on superficially treated Ti-6Al-4V give evidence of the main effect of residual stresses. In the case of superficial treatments, tests are generally made with premachined pieces so to keep the diffusion layers. Thus, residual stresses due to machining disappear and the lack in structural changes during cooling prevents for the development of new internal stresses.

So, we think that a good treatment for both fatigue and friction uses may be a treatment with chemically unstable layers and for this, we may suggest nitrogen.

Another way of introducing beneficial residual stresses may be structural changes in the alloy matrix. We are studying the possibility of a transformation during fatigue straining. IMAM and GILMORE (3) recently showed that specimens treated at 900°C and water quenched have good fatigue properties and this improvement corresponds to 50 pct α', 25 pct α and 25 pct α". For low cycle fatigue tests, we are following the stiffness of the matrix, for several temperatures of treatment, so to notice strain induced phase transformations and delays in fatigue cracking.

In conclusion, fatigue behavior of superficially treated Ti-6Al-4V alloys is mainly to be analyzed through the nature of residual stresses. Pre-existing stresses are needed to avoid strong decreases in fatigue limits but it seems that transformations that may occur in the superficial layers and in the matrix are to be searched.
References

Acknowledgements
The authors acknowledge Dr. Champin (Ugine) and Wheelabrator-Allèvard for their helps in the preparation of specimens. They also thank E.T.C.A. - DRET for their helps in continuing this study.