MICROSTRUCTURE AND FRACTURE OF TITANIUM ALLOYS

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Abstract
The following work deals with microstructure and deformation investigation of titanium Ti-1.5Al-1Mn (OT4-1) and Ti-6Al-4.5V (VT6) alloys prepared from powders produced by the hydro-dehydrogenation method (HDH), while the initial raw material was titanium sponge. Compaction of the titanium powders was carried out by hot isostatic pressing (HIP) and with the acquired samples there was study as to their physical and mechanical properties, in their relationship to structure and mechanical deformation. Mechanical properties, especially the strength characteristics of the materials produced, reached the property levels of alloys produced in the classic conventional style (KON). A greater difference appeared in plastic properties which with the HIP samples were very low, and we presume that in the resulting coarse-grained structure there is increased gas content and impurities at the edges of the grain, as well as the existence of pores. Fractographic observation of fractured surfaces confirmed that, through the dominant mechanisms of deformation, there appears a ductile transcrystalline mechanism, and to a lesser extent were identified mechanisms of intercrystalline division and a ductile deformation with dimple morphology.

Keywords: powder metallurgy, titanium alloys, HIP, mechanical properties, fracture

INTRODUCTION
Titanium alloys are classified as α, pseudo-α, α+β and β-alloys. Pure titanium is a polymorphic metal and has two allotropic modifications and that is α-phase with a hexagonal lattice and β-phase with a body centred cubic lattice. Alloying elements are counted α or β type stabilisers. Among the α-stabilisers are included Al, O₂, N, C, Sn, Zr, and Si, and Cu, V, Cr, Mn, Fe, Co, Ni, Mo are the β-stabilisers. Titanium alloys are marked by a relatively low specific weight, relatively high strength, good wear resistance and brittle fracture resistance, good refractoriness, corrosion resistive, and chemical stability in aggressive conditions. The wide ranging use of these materials in practice however, is restricted by the energy requirements for manufacturing, and for this reason their use is limited to aviation, and the aerospace and chemical industries. These materials find a useful role in specific power industry applications, for example, in rotary turbines or within medical science as part of implants for instance.

At the present time, many ways to produce titanium powder are known of [1], but the method of hydro-degeneration (HDH) is marked mainly by the fact that it may process titanium scrap or sponge. Through PM technology, a reduction in labor-intensiveness and
a better use of material has been achieved. The disadvantage of the material is a high affinity to oxygen, nitrogen, sodium and carbon [2,3].

The objective of the work is the analysis of the microstructure and fracture of experimental titanium alloys prepared by powder metallurgy.

**EXPERIMENTAL MATERIAL AND TESTING METHODS**

For the experimental, titanium powders were produced by the HDH method. Technically pure non-alloyed powder was produced from titanium chips and pre-alloyed titanium powder from the residual scrap acquired during the mechanical processing of the scrap of alloys VT6. The basis of the HDH method is that the washed chips were dried out in a vacuum of 1Pa at a temperature of 90-100°C, and had to undergo hydrogenation in a silite retort furnace at a temperature of 500-600°C for a period of 60 min in a hydrogenous atmosphere. The obtained sponge was then ground in a bearing mill in an argon atmosphere, and dehydrogenation was carried out in a vacuum of $10^{-3}$ Pa for a period of 4 h at a temperature of 820°C. The same approach was employed also during the production of Ti-powder from titanium remnants.

Two types of titanium powder materials were prepared for the experimental program, that is:

a) blend system: a mixture from the remainder from the technically pure Ti-powder, to which was added the elementary powders of Al, Mn corresponding to a ratio matching the chemical composition of the alloy OT4-1 (Ti-1.5Al-1Mn),

b) prealloyed powder alloy VT6 (Ti-6Al-4V).

Gases and granulometric composition were adjusted for both materials there. The powder compaction was realized by HIP at a pressure of 150 MPa, and at a temperature of 1100°C for a period of 2 h. From both materials there were produced samples for static tensile tests, and samples for individual analyses.

For each material a metallographic and fractographic analysis was carried out, and total porosity was measured. From both alloys thin foils were prepared for transmission electron microscope observation using the JEM-100CX microscope. Thin foils of were electrolytically polished on TENUPOL.

**RESULTS**

For Ti-powder and pre-alloyed VT6 powder the assigned gas contents were O$_2$, N$_2$, H$_2$, Fig.1 and granulometric composition as well, Fig.2.

In Table 1 there are presented mechanical properties of titanium alloys OT4-1 and VT6 after HIP and KOM.

<table>
<thead>
<tr>
<th>Materials</th>
<th>Technology</th>
<th>Ultimate tensile strength Rm [MPa]</th>
<th>Yield Strength Rp0.2 [MPa]</th>
<th>Ductility A$_5$ [%]</th>
<th>Contraction Z [%]</th>
<th>Total porosity Pc [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>OT4-1 HIP</td>
<td>680</td>
<td>68</td>
<td>3</td>
<td>12</td>
<td></td>
<td>1</td>
</tr>
<tr>
<td>OT4-1 KOM</td>
<td>720</td>
<td>650</td>
<td>14</td>
<td>38</td>
<td></td>
<td>-</td>
</tr>
<tr>
<td>VT6 HIP</td>
<td>1020</td>
<td>950</td>
<td>3</td>
<td>9</td>
<td></td>
<td>1</td>
</tr>
<tr>
<td>VT6 KOM</td>
<td>1100</td>
<td>1000</td>
<td>11</td>
<td>39</td>
<td></td>
<td>-</td>
</tr>
</tbody>
</table>
Metallographic analyses indicated that the microstructure of the alloy OT4-1 (HIP) was heterogeneous, non-polyedric, and was comprised of the $\alpha$-phase grains and a small 5% flat share of grains of the $\beta$-phase. Porosity did not exceed 1% and present oval pores were very fine. During the metallographic cutting, EDX microanalysis identified the following elements: Ti, Al, Mn, O and C, of which the linear analysis is documented in Fig.3. The transmission electron microscope analysis, that the sub-structure was left over from the $\alpha$-grains and isolated cases were identified as $\beta$-grains Fig.4. In the sub-structure of alloy OT4-1 were monitored oxidized particles at the edge of the grain at a size of 0.1 $\mu$m, and in Figs.5 and 6 were observed Ti$_3$Al particles which were identified by diffractography. The fracture surface of the OT4-1 samples after HIP consisted of the facets of transcrystalline cleavage and, in isolated cases, intercrystalline separation, Fig.7.

Fig.1. Gas content in Ti and VT6 powder.

Fig.2. Granulometric composition of powders Ti and VT6.

Fig.3. Microstructure of the alloy OT4-1.

Fig.4. Transmission electron microscopy of the alloy OT4-1.
Metallographic analysis confirmed that the structure of the alloy VT6 (HIP) is created by $\alpha + \beta$ phase Fig.8, while $\alpha$-phase forms the matrix and $\beta$-phase represents light irregular formations and comprises approximately 10% of the surface area. In Figure 8 they are identified by linear analysis. The substructure of alloy VT6 is created by $\alpha$ and $\beta$-grains as presented in Fig.9. From the fractographic perspective it may be stated that the dominant mechanism was transgranular ductile mechanism transcrystalline cleavage, while there was transcrystalline cleavage in the $\alpha$-phase and the fracture was caused by ductile bridges or dimple morphology of the $\beta$-phase, Fig.10.
DISCUSSION

Titanium powders produced by the HDH method are, from the morphological aspect, characterized by emerging from the fracture of larger particles and individual flat surfaces, and have a morphological cleavage facet. Chemical analysis of gases showed that with Ti-powder, lower values for O₂, N₂ and H₂ content were set than for pre-alloyed VT6 powder, see Fig.1. Granulometric analysis showed that the Ti-powder was significantly finer and its fraction under 0.1 mm represented 86% of total fraction, as for the pre-alloyed powder VT6 it was only 32%.

Since the technique of compaction was equal for both materials (HIP), it was possible for the mechanical property values to be compared with each other, while at the same time aligning them to conventional alloys. The alloy OT4-1 (HIP) showed significantly lower strength properties than the pre-alloyed VT6 (HIP), while the plastic properties of both were low, to approximately the same extent. It is a result of the lower ratio of alloying elements in the OT4-1 alloy and the related structural composition. OT4-1 is a pseudo-α alloy, which means that the contents of the β-phase do not reach 5%, as long as the alloy VT6 is double-phase (α+β), and in our case, the β-phase represents approximately 10% of the flat area. These alloys produced (via) through PM in comparison to the conventional ones displayed a very small difference as to strength properties, but as to the plastic properties they markedly differed. Higher strength properties of HIP samples were achieved as a result of the fine-grained structures of the conventional materials, because for them there had been applied thermo-mechanical processing, whereas in the samples produced after HIP, that technological operation was absent. Low plastic properties of the samples after HIP were due to the coarse-grained microstructure as in conventional alloys, with higher gas content, segregation of impurities, and undesired elements detected on the edge of the grains as supported by analyses of the substructures. Although measuring the entire porosity showed that, by the process of HIP, the porosity was almost eliminated less than 1%. However, within the structure were found identifiable pores, that being documented on the photograph of the alloy OT4-1, Fig.3.

From the viewpoint of fractography, fractured surfaces of the OT4-1 alloy may be considered as a ductile fracture controlled by the cleaving of the matrix, Fig.7, outlining the fractured surfaces in a non-polyedric lattice structure, of which the basis is comprised of α-phase materials with a small mutual disorientation. The entire process of the deformation of a non-polyedric structure will be explained in the following approach; during the deformation process in some places one arrives at the emergence of disruption or cracking,
which does not spread out over the entire grain, but stops before the first obstacle, which may be a sub-boundary or a structural $\beta$-phase formation, stopping it. At the tip this brings about an additional concentration of tension, which then contributes to the repeated nucleation breakage at the same level in the neighbouring sub-grain. Bridging the cracks happens through a ductile mechanism, i.e. a complete reduction of bridges between neighbouring fissures. The crack which we have called a ductile break with structurally directed trajectory is then controlled by the fissure of the matrix. Also observed with the same material were locations of intercrystalline division, which made for a segregation of impurities and undesired elements on the edges of the grains and locations with pit morphology. The size of the dimple was about 0.6 up to 1 $\mu$m.

VT6 alloys are marked by a fine polyedric structure created by $\alpha + \beta$ phase, and there were also pores identified in the structure whose volume was less than 1%. The substructure consisted of $\alpha$-grains, among which and in radial shapes appeared $\beta$-grains of which the flat area did not exceed 10%. Also with the polyedric alloy VT6 there was observed a granular ductile fracture mechanism of the $\alpha + \beta$ phase. This means that during work there occurred a transcrystalline fracture of the $\alpha$-phase, and the cleavage finished by a ductile deformation of the $\beta$-phase. Deformation of the $\beta$-phase had the appearance of a ridge, or perhaps lines, but with larger volumes of $\beta$-phase there occurred a pitted deformation, Fig.10.

**CONCLUSIONS**

Shown in practice was the possibility of producing powders from the chipped remainder of titanium alloys, or titanium remnants by the HDH method, and technological approaches for their productions were proposed. The strength properties of the samples (HIP) achieved the levels of conventional alloys, but as for plasticity it was significantly lower. With the OT4-1 alloy there was observed a ductile fracture controlled by a break in the matrix and localities with intercrystalline separation. From the point of view of fractography, the VT6 alloy was distinguished by ductile transcrystalline cleavage.

**REFERENCES**