

POLISH ACADEMY OF SCIENCES - COMMITTEE OF MATERIALS SCIENCE SILESIAN UNIVERSITY OF TECHNOLOGY OF GLIWICE INSTITUTE OF ENGINEERING MATERIALS AND BIOMATERIALS ASSOCIATION OF ALUMNI OF SILESIAN UNIVERSITY OF TECHNOLOGY

Conference Proceedings

ACHIEVEMENTS IN MECHANICAL & MATERIALS ENGINEERING

Influence of thermal shock on titanium alloy microstructure*

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Deformation followed by recrystalise annealing are usually used processes for grain refinement. Some literature sources reports that heat treatment followed by no plastic deformation is applicable too. There are known many kinds of annealing heat treatment used for titanium alloys: stabilise, homogenise, recrystalise and stress reveals annealing. In our case was for grain refinement used method, known in sources as thermal shock treatment.

1. RESOLVE STAGE

We research various effect of energy for obtaining optimal utility properties of titanium alloy Ti6Al4V, [1] in our laboratories. Microstructure patterns of certified alloy Ti6Al4V remelted in laboratory plasma furnace at solving workplace are in Fig. 1 and Fig. 2. [1].

1.1 Thermal shock heat treatment

Some information on treatment of metals by thermal shocking is briefly reported [2]. Heating up in temperature range of phase transformation $\alpha \leftrightarrow \beta$ is required for treatise of titanium alloys. All process shall be expedient to do in vacuum or inert atmosphere. There is known from literature, that treatise temperature range 850°C to 950°C could create polygonal substructure. Repeated microstructure changes, it means repeated transformation in $\alpha \rightarrow \beta \rightarrow \alpha$ series generate internal stresses and polygonal deformation inside the grains. The "PANAMA" microstructure pattern will be obtained. The α - phase lamellas disintegrate to subgrains, β - phase lamellas fragment in appropriate way too.

Work [2] reports, that advanced thermally stable subgrain microstructure with small disorientation angle (about 3°) will be created in double phase titanium alloy after treatment above. Work also shows, that referred treatment can be effective way to reinforce material, then provide toughness, plasticity and crack resistance. Peculiar microstructure, comprised by colonies, is created in the cast or usual heat treated microstructure of Ti64 alloy. Colonies are regions within lamellar former β - phase with same orientation. Colonies

^{*} Authors participate in the GRANT VEGA SR No. 1/7170/2000. Authors participate in the CEEPUS No PL-013/03-04 project headed by Prof. L.A. Dobrzański.

are generated during the cooling down process from β - phase by the cooling speed inducing lamellar morphology.



Figure.1 Microstructure of sample A - grain Figure.2 Microstructure of sample A ($\alpha + \beta$) boundaries

2. PLASMA METALLURGY RESULTS

Results of microstructure analysis of certified titanium alloy Ti-6Al-4V, prepared in laboratory plasma furnace at solving workplace follow. There were samples marked as A and I to V. Microstructure analysis, x-ray diffraction and Vickers hardness HV 30 measurement were done. The samples has been chemical – mechanical polished in OP-S solution, see Tab. 1. Sample A has been polished in 60% OP-S + 40% H₂O₂ solution, samples I to V has been polished by the diamond cream with granularity 6 μ m and 4 μ m, followed by OP-S 70% OP-S + 30% H₂O₂ solution. Etching dilution was 1,5 ml HF + 3 ml HNO₃ + 10 ml H₂O and etching time was 3 sec.

Sample I was affected by the 5 cycles and its bigger part of microstructure area is not changed casting microstructure, but orientation has been changed to [002]. Microstructure of sample V, affected by the 25 cycles, is noticeable changed with orientation [101] and former microstructure was eliminated in total. It follows from experiments that increasing number of thermal cycle eliminate microstructure orientation. Phase α ' has been detected in the sample I by the x-ray analysis, but not in the rest samples.

The presumed sample hardness increase due to thermal shock was confirmed. Hardness of samples is increasing by the number of applied cycles.

3.1 Tensile strength and hardness tests

Bars for tensile strength test were made from ingot prepared in plasma furnace. Measurement has been done by the INSTRON machine. Bar was standard type with dimensions $d_0 = 5 \text{ mm}$ and $l_0 = 23 \text{ mm}$ respectively. Mean tensile strength was $R_m = 1196$ Mpa, ductility $A_5 = 6,95\%$ and Z = 4,22%. Measured values shows, that certified cast alloy Ti6Al4V remelted in plasma furnace has tensile strength and ductility comparable with literature quoted values. In Table 1 has been presented Vickers hardness results after thermal shocks.

1/5	11/10	III / 15	IV / 20	V/25
319	355	343	395	426
331	349	353	413	428
315	364	368	413	426

 Table 1

 Vickers hardness of analysed samples after thermal shocks

3.2 Microstructure analysis

Sample A microstructure pattern of plasma furnace remelted certified alloy Ti6Al4V is in Fig.1 and 2. There is seen that morphology of alloy is lamellar. The α - phase stabilised by aluminium is bright. Aluminium penetrates into α -phase and reinforces its solid solution. Maximum content of aluminium in alloy is 6 wt. % and aluminium eliminates creation of Ti₃Al phase which is decreasing ductility and corrosion resistance. Along grain boundaries of α - phase is located β - phase, seen dark in figures. The β phase is containing vanadium that is lowly dissolvable in α - phase. Microstructure of researched alloy is double-phased (α + β). Heterogeneity of alloy with clear grain interfaces is visible in this pictures.

Microstructure patterns of thermal shock treatment alloy Ti6Al4V samples 2 to 4 are in Fig.3 to 7. Samples are different in applied thermal shocks number. Structure of these samples is lamellar. Fine homogenous structure due to thermal shock treatment was presumed. Structure is homogenised in compare to A1 and A2 samples. Mean number of lamellas is increasing with number of heat treatment cycles, see Tab. 2. It means, that microstructure was refined.



Figure.3 Microstructure of sample I after 5 thermal shocks



Figure.4 Microstructure of sample II after 10 thermal shocks



Figure.5 Microstructure of sample III after 15 thermal shocks



Figure.6 Microstructure of sample IV after 20 thermal shocks



Figure.7 Microstructure of sample V after 25 thermal shocks

3.3 X-ray diffraction analysis

X-ray diffraction analysis was performed on I to V samples. We can conclude, that orientation of sample II is in direction of [100] and samples III and IV has orientation in direction of [112], Fig.8. Sample shows advanced direction annihilation. Cast structure in samples IV and V was eliminated in comparison of I, II and III samples. Big tensions / stresses were generated in *all* samples.



Figure.8 Diffraction patterns of analysed alloys

4. CONCLUSION

Analyse of experiments shows, that very pure titanium alloy can be prepared in laboratory plasma furnace. It means, that interaction of melting alloy with carbon, oxygen, nitrogen and hydrogen was absolutely eliminated during preparation of castings and ingots. There was proved significant influence of thermal shocks to change of cast microstructure of Ti6Al4V. Polygonal, homogenous structure was not even reached but way presented above can assure more homogenous microstructure. These results must be supported by notch toughness test.

ACKNOWLEGMENTS

The autors acknowledge the Scientific Grant Agency of Ministri of Education of Slovak Republic (VEGA SR No. 1/7170/00) for the financial support of this work

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